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Washington, DC 20460

EPA/540/8-89/012  
December 1988

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Superfund

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# User's Guide to Contract Laboratory Program



EPA/540/8-89/012  
December 1988

USER'S GUIDE TO CONTRACT LABORATORY PROGRAM

**U.S. Environmental Protection Agency**  
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U.S. ENVIRONMENTAL PROTECTION AGENCY  
OFFICE OF EMERGENCY AND REMEDIAL RESPONSE  
401 M STREET SW

Washington, DC 20460

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Environmental Protection Agency  
for the  
United States  
Department of the Interior

## **FOREWORD**

This document has been prepared by the CLP Sample Management Office specifically for the guidance and direction of program clients. The organic and inorganic analytical program descriptions herein outline the requirements and analytical procedures of the new CLP protocols developed from technical caucus recommendations. These protocols were implemented into CLP analysis contracts in 1987 (inorganic) and 1988 (organic). Other analytical programs, procedures and documentation described herein reflect the status of the program as of December 1988. Critical information for CLP samplers and user groups is contained in Chapter III and Appendix D. This information should be distributed to all contractors collecting samples for the CLP and to each user group of the EPA and of the States. For further information on the CLP or to obtain additional copies of the User's Guide, contact the Sample Management Office at 703/557-2490 or FTS 557-2490.

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## **CHAPTER I**

### **BACKGROUND AND INTRODUCTION**

### **A. CLP Objective and Orientation**

The Contract Laboratory Program (CLP) supports the Environmental Protection Agency's (EPA) Superfund effort, originally under the 1980 Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA) and presently under the 1986 Superfund Amendments and Reauthorization Act (SARA). The CLP provides a range of state-of-the-art chemical analytical services of known quality on a high volume, cost effective basis. The CLP is structured to provide legally defensible analytical results for use in supporting ongoing Agency enforcement actions or other requirements of the user community. Therefore, a level of quality assurance and documentation appropriately designed for the intended purposes of the data has been incorporated into all aspects of program activities.

Client orientation is a key factor in the design and application of all CLP services and responses. The CLP supplies analytical services in direct response to requests from the EPA Regions, the primary users of the program. Recently, states and other Agency programs have also become part of the CLP user community.

The ongoing CLP objective is to develop, manage and improve its analytical programs in support of all Superfund requirements. This objective is accomplished by continually increasing analytical capacity and adjusting analytical program requirements and related support services to better meet Agency needs.

### **B. CLP Structure**

CLP services involve numerous Agency programs, contractors and other groups throughout the country. These organizations are identified and their role in the program described in the following sections. The following figure, "Interrelationships of Program Principals," illustrates the interaction of these groups in CLP operation. In addition, a directory listing addresses and telephone numbers of key program personnel is located in Appendix B.

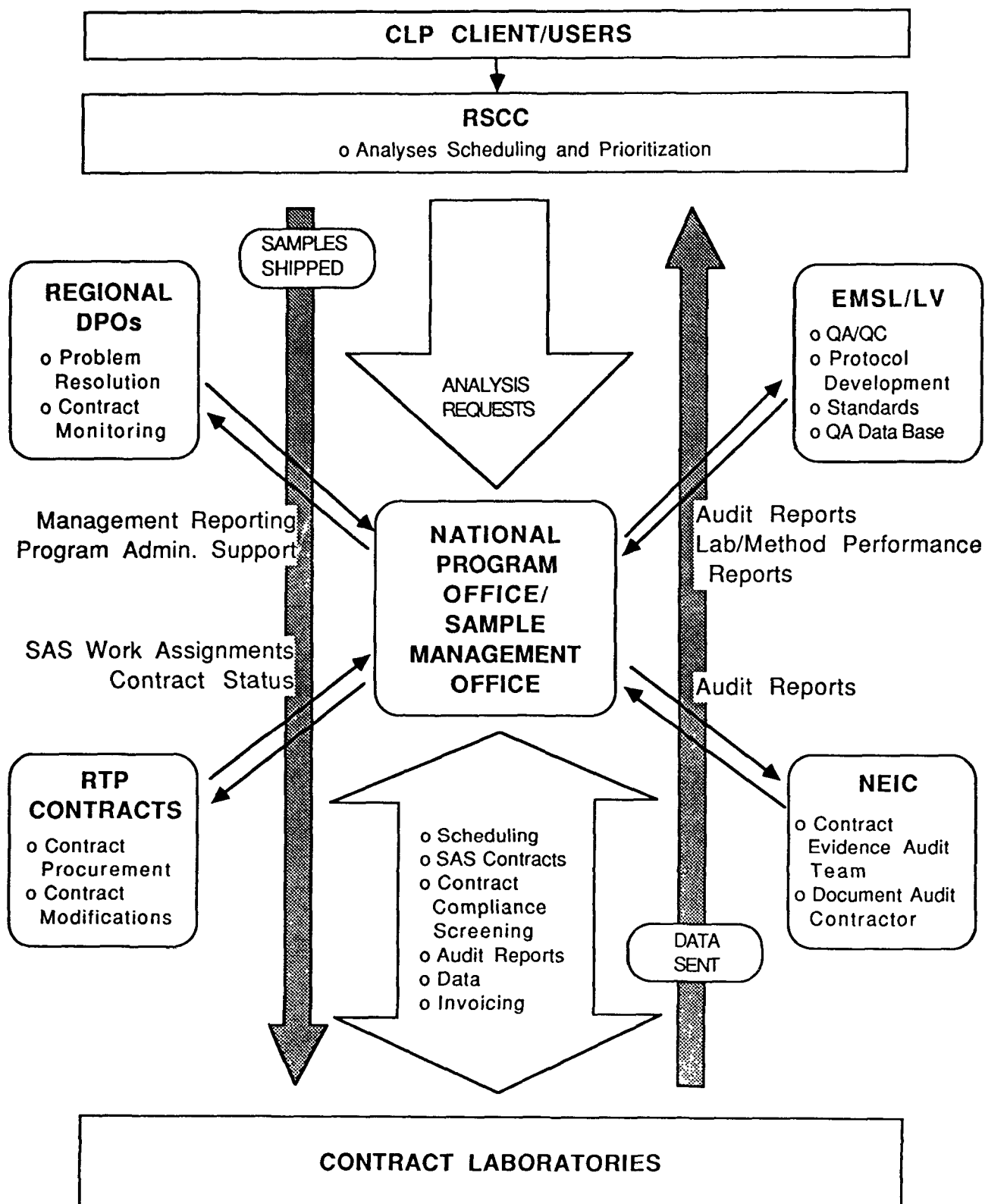
#### **1. Program Management**

##### **a. National Program Office**

The CLP is directed by the National Program Office (NPO), in EPA Headquarter's Analytical Operations Branch (AOB), Hazardous Site Evaluation Division (HSED), Office of Solid Waste and Emergency Response (OSWER), located in Washington, DC. The NPO is comprised of the AOB Branch Chief; National Organics and Inorganics Program Managers; a Regional Operations Chief; a Quality Assurance Coordinator; and Organics, Inorganics and Dioxin Technical Project Officers.

NPO responsibilities include: overall management of the CLP in terms of program objectives; expansion and interface with clients and other groups; policy and budget formation and implementation; development and administration of CLP analytical and support services contracts; development and technical review of analytical protocols; review of Special Analytical Services subcontracts and CLP-generated laboratory data; monitoring and formal evaluation of analytical and support contractors; and direction of CLP quality assurance in coordination with overall OSWER quality assurance activities.

## INTERRELATIONSHIP OF PROGRAM PRINCIPALS



The National Organics and Inorganics Program Managers (NPMs), in addition to directing organics and inorganics section staff, are responsible for the formulation of CLP policies and direction. By communicating with Regional and Agency communities on a continuing basis, the NPMs keep all parties apprised of program activities and receive input on program effectiveness. The NPMs also direct annual technical caucuses for the purpose of reporting initiatives and progress of the past year.

The Regional Operations Chief directs a staff responsible for the Sample Management Office contract, the Environmental Services Assistance Teams contracts, the Sample Bottle Repository contracts, and the Shipment Management contract. In addition, the Regional Operations Section manages the supply and demand between CLP capacity and client needs, and provides budget support and administration.

The Quality Assurance (QA) Coordinator manages all aspects of program application of QA procedures. The QA Coordinator works closely with Office of Research and Development's Environmental Monitoring Systems Laboratory in Las Vegas (ORD EMSL/LV) in administering and improving the QA program. The QA Coordinator interacts with the Project Officers in refining and updating analytical method QA and communicates with the Regions and other program users to resolve QA issues related to analytical data. For purposes of QA procedures review and guidance development, the QA coordinator conducts volunteer workgroups throughout the year.

The Technical Project Officers (POs) are responsible for technical program decisions, contract monitoring and contractor performance evaluation. On a daily basis, the POs work closely with the Regional Deputy Project Officers and contract laboratories to resolve technical issues. The POs also direct the ongoing effort to improve contract language and analytical methodologies. For the purposes of CLP protocol review and method development, the POs conduct volunteer workgroups throughout the year.

**b. Sample Management Office**

The contractor-operated Sample Management Office (SMO) provides management, operations and administrative support to the CLP. The primary objective of SMO is to facilitate optimal use of program analytical resources. SMC activities fall into the following areas: 1) sample scheduling and tracking, 2) Contract Compliance Screening, 3) Special Analytical Services subcontracting, 4) laboratory invoice processing, 5) maintenance of CLP records and management reporting, 6) procurement/IFB development and Statement of Work production, 7) coordinating CLP meetings and conferences and 8) NPO management, technical and administrative support.

SMO routinely receives Regional analytical requests, coordinates and schedules sample analyses, tracks sample shipment and analyses, receives and checks data for completeness and compliance, processes laboratory invoices, and maintains a repository of sampling records and program data. In response to client requests for nonroutine types of analyses, SMO subcontracts for Special Analytical Services (SAS), scheduling and tracking for SAS efforts as outlined above. SMO maintains a comprehensive database of CLP services, performance and utilization in order to generate a variety of management and user reports.

c. Office of Research and Development, Environmental Monitoring Systems Laboratory/Las Vegas

ORD provides program QA support through EMSL/LV. EMSL/LV assists in performing preaward and postaward onsite laboratory evaluations; prepares performance evaluation (PE) samples for preaward and postaward evaluations of laboratory performance; evaluates preaward and postaward PE sample data; performs QA audits on CLP-generated data; and assists in the evaluation and development of CLP analytical methods and protocols. Additionally, EMSL/LV operates the program's QA database to conduct program and laboratory trend analyses used in developing and updating contract quality control criteria.

d. National Enforcement Investigations Center

The National Enforcement Investigations Center (NEIC) advises the NPO in defining and applying program enforcement requirements. NEIC-developed sample custody procedures, chain-of-custody records, sample tags, and custody seals are utilized in the CLP to maintain the validity of sample analyses for supporting Agency enforcement actions. NEIC routinely performs evidence audits of contract laboratories and generates sample profiles used in Agency enforcement litigation. A description of the enforcement support provided by NEIC appears in Chapter IV, Section E.

## 2. Regional Program Support

The Regions play an integral role in program activities, both as the primary CLP user and as a key part of analytical program management. The decentralization of program responsibilities to the Regions is an effective means of directing program operations nationwide. Extended Regional participation in the program has and will continue to increase the program's responsiveness to Superfund requirements.

a. Regional Deputy Project Officers

In 1984, Regional Administrators appointed a CLP Technical Deputy Project Officer (DPO) for each Regional office. Under the direction of the NPO, the Regional DPO monitors the contract laboratories located in the Region. The DPO works closely with the PO in responding to identified problems in laboratory operations and participating in laboratory onsite evaluations.

b. Regional Sample Control Centers

In 1984, each Region established a Regional Sample Control Center (RSCC) to centralize scheduling of CLP sample analyses within the Region. The RSCC is comprised of one or more individuals with one individual named as the primary RSCC. The RSCC is responsible for coordinating the level of Regional sampling activities to correspond with the monthly projected demand for analytical services. The primary RSCC makes final determinations regarding Regional analysis priorities when conflicts occur. The RSCC routinely places all Regional requests for CLP analyses, coordinates with SMO during sampling and sample shipment, and resolves any problems concerning the samples. The RSCC also serves as the central point of contact for questions concerning Regional sampling efforts.

c. Regional/Laboratory Communication System

In 1983, the NPO established a system by which the Regions and contract laboratories can communicate in the most timely and direct manner possible. In this communication system, designated Regional communication contacts routinely call designated laboratory communication contacts to resolve technical questions concerning program data. This communication link also benefits the laboratory by providing direct feedback on its data product.

**3. Clients/Users**

a. EPA Regions

The ten EPA Regions are the primary clients of the CLP. As previously described, each Region has established an RSCC that schedules all Regional CLP analysis requests. The RSCC balances Regional sampling with allocated numbers of CLP sample analyses available each month and prioritizes the Region's analytical workload when conflicts occur. RSCC personnel coordinate closely with SMO throughout Regional sampling events, assisting in tracking sample shipments to the laboratory and resolving any problems that arise. The RSCC also processes analytical requests from state or other program users that are located in the Region's geographical area.

b. States

Under RCRA - CERCLA Cooperative Agreements, any state undertaking initial site investigations and entering into cooperative agreements with the Government for clean up of local waste sites can utilize CLP services. States must access CLP analytical services through the RSCC. Data packages are also distributed to states through the RSCC.

c. NonSuperfund Clients

Program services are available to support nonSuperfund clients. NonSuperfund analyses and other support are provided by the CLP through transfer of funds from the nonSuperfund program to the CLP. NonSuperfund clients currently include other government agencies and EPA programs, such as the Office of Acid Deposition, the Office of Solid Waste, the Office of Water, and the Resource Conservation and Recovery Act.

**4. Analytical and Support Services Contractors**

a. Contract Analytical Laboratories

The CLP's analysis contractors come from the nationwide community of chemical analytical laboratory facilities. To become part of the CLP, laboratories must meet stringent requirements and standards for equipment, personnel, laboratory practices, and analytical and quality control operations. Firm, fixed price contracts are awarded to the lowest responsive, responsible bidders through the Government's Invitation for Bid (IFB) process. Before a contract is awarded, low priced bidders must successfully analyze performance evaluation samples and pass a preaward laboratory audit. After contract award, laboratories are closely monitored to assure compliance with the terms and conditions of the contract. Details of preaward and postaward evaluations are addressed in Chapter V.

**b. Sample Bottle Repository**

In 1982, the NPO established the Superfund Sample Bottle Repository program in order to provide a common source of clean, quality control tested sample containers for samples processed through the CLP. The objective of the Repository program is to eliminate the potential of bottle contamination that would affect the validity of sample data. The contractor-operated repositories serve as a central source for several types of precleaned sample containers which are routinely utilized by Regional and contract personnel performing Superfund sampling activities. Containers are also available through the Repository program for nonSuperfund sampling activities such as those under the National Surface Water Survey and the Resource Conservation and Recovery Act. Repository services are detailed in Chapter IV, Section A.

**c. Shipment Management Program**

The Shipment Management program was created by the NPO in 1988 to provide a consistent means of tracking the various shipping accounts established for CLP use. The Shipment Management Contractor is responsible for establishing, maintaining and monitoring the shipping accounts for the transportation of sample containers, sample coolers, contract compliance screening results and other items requested by the NPO. Further information on the Shipment Management program is provided in Chapter IV, Section B.

**d. Environmental Services Assistance Teams**

In 1985, the NPO initiated the concept of Environmental Services Assistance Teams (ESAT) to provide a wide range of technical, management and other related resource support for Superfund and nonSuperfund Agency programs. ESAT contractors assist EPA Headquarters and the Regions in the following task areas: 1) analytical support, 2) data review, 3) logistical and administrative support, 4) quality assurance/quality control support, 5) management and reporting, and 6) other task-related activities. ESAT services are detailed in Chapter IV, Section C.

## **CHAPTER II**

### **DESCRIPTION OF ANALYTICAL SERVICES**



The CLP provides routine and specialized analytical services to support a variety of Superfund sampling activities. These activities range from those associated with the smallest preliminary site investigation to those of large scale, complex remedial, monitoring and enforcement actions. In response to the increasing analytical demands of Regional clients, the CLP has continually expanded its capacity for standardized analyses through frequent contract solicitations. On the average, the CLP provides over 6,000 sample analyses per month through its routine and specialized analytical services programs. The CLP will continue to adjust analytical capabilities and capacity in response to client needs.

The CLP operates the following analytical programs:

- o Organic Routine Analytical Services (RAS),
- o Volatile Organic RAS,
- o Inorganic RAS,
- o Dioxin RAS, and
- o Special Analytical Services (SAS).

In the future, many other analytical programs will be included under RAS:

- o High Concentration Organics
- o High Concentration Inorganics
- o Organics Low Concentration (Drinking Water)
- o Inorganics Low Concentration (Drinking Water)
- o GC/EC Pesticides/Aroclors
- o Fast Turnaround GC Screen Organics
- o Dioxins/Furans
- o ICP/MS (method to be included in Inorganic RAS), and
- o Microwave Digestion (method to be included in Inorganic RAS).

Laboratories operating under firm, fixed-price contracts with the EPA provide routine analytical services to Superfund clients. NonSuperfund clients can also access RAS programs once special funding arrangements have been made.

The SAS program provides nonstandardized analytical services to Superfund and nonSuperfund clients for organics, inorganics, dioxin and other compounds in a variety of matrices. SAS services are offered to meet specific analytical requirements which do not fall under RAS programs and are solicited through individual fixed-price subcontracts awarded to qualified laboratories.

The following tables outline the services available under the CLP's RAS and SAS programs. The client should carefully consider the provisions of each CLP analytical program during the planning stages of a sampling event to determine the applicability of the analysis to user needs. For detailed analytical information, users are instructed to consult the Region's Master Copy Statements of Work under which CLP RAS laboratory contractors operate.

## MENU OF ROUTINE ANALYTICAL SERVICES

CATEGORY	ORGANIC ANALYSIS	VOLATILE ORGANIC ANALYSIS	INORGANIC ANALYSIS	DIOXIN ANALYSIS
SAMPLE MATRICES	Low & Medium Concentration Water & Soil/Sediment Samples	Low & Medium Concentration Water & Soil/Sediment Samples	Low & Medium Concentration Water & Soil/Sediment Samples	Low & Medium Concentration Water & Soil/Sediment Samples
COMPOUNDS IDENTIFIED & QUANTIFIED	Target Compounds & Library Matches of 30 Highest Compounds (In the ppb range)	Volatile Target Compounds & Library Matches of 10 Highest Compounds (In the ppb range)	Metals & Cyanide (In the ppb range)	2,3,7,8-TCDD (In the ppb range)
DATA DELIVERY	Thirty-five Days	Fourteen Days	Thirty-five Days	Twenty-one Days (Routine) Sixteen Hours (Rapid Turnaround)
ANALYTICAL PROCEDURES	GC/MS Analysis (VOA, BNA) GC/EC Analysis (PEST) Following Sample Preparation/Extraction	GC/MS Analysis Following Sample Preparation/Extraction	Flame/Flameless & Cold Vapor AA; ICP & Colorimetric Analysis	GC/MS Analysis by FSCC Following Solvent Extraction/Clean Up
QA/QC SUMMARY	Surrogate Spike In Each Sample; Matrix Spike Duplicate Per Sample Delivery Group* For Each Matrix and Concentration On Per-Fraction Basis	Surrogate Spike In Each Sample; Matrix Spike Duplicate Per Sample Delivery Group* For Each Matrix and Concentration On Per-Fraction Basis	Matrix Spike & Duplicate Per Sample Delivery Group* For Each Matrix and Concentration	Matrix Spike & Duplicate Per Batch of 24 Samples or Less

\* A Sample Delivery Group is a group of samples within a Case received over a period of fourteen days or less and not exceeding twenty samples. A Case designates a group of samples collected at one site or geographical location during a specific finite period of time.

**MENU OF SPECIAL ANALYTICAL SERVICES****RAS Plus SAS Category**

- o Fast Turnaround Analysis by RAS Organic, Inorganic or Dioxin IFB Protocol
- o RAS Organic Analysis with Additions/Adjustments to IFB Protocol
- o RAS Inorganic Analysis with Additions/Adjustments to IFB Protocol
- o RAS Dioxin Analysis with Additions/Adjustments to IFB Protocol

**All SAS Category**

- o Organic Analysis Per Non-RAS Protocols, Matrices, Compounds
- o Inorganic Analysis Per Non-RAS Protocols, Matrices, Compounds
- o Dioxin Analysis Per Non-RAS Protocols, Matrices, Compounds
- o Organic and Inorganic High Concentration Sample Preparation and Analysis
- o Special Topics Analysis (As Requested)

**NOTE:** The client is responsible for designating IFB method adjustments for "RAS Plus SAS" requests and for supplying suitable analytical protocols for "All SAS" requests. Additionally, the client must provide quality assurance/quality control procedures and criteria, and must specify data delivery schedules. All information must accompany the client's request for SAS services.

Routine analytical services apply to the analysis of water and soil/sediment samples. Samples for analysis should be single-phase and homogeneous. Sample matrices other than water or soil/sediment are processed through the SAS program.

Organic and inorganic RAS contract methods determine low to medium concentrations of organic target compounds and inorganic target analytes, respectively. The sampler identifies low and medium levels of concentration in the field to determine sample collection volume and packaging and shipment procedures. Low level samples are considered to be those collected off site in areas where hazards are thought to be significantly reduced by normal environmental processes. Medium level samples, where a compound or element may comprise as much as fifteen percent of the total sample, are most often those collected onsite in areas of moderate dilution by normal environmental processes. The contract laboratory performs preliminary characterizations to determine the appropriate analytical protocol (low or medium) to be used.

Required sample volume and container types used for sample collection for RAS analyses are detailed in the following sections and are illustrated in Appendix D. Cleaned, quality controlled sample containers are available through the Sample Bottle Repository as described in Chapter IV, Section A. Containers provided by the Repository may also be utilized in SAS projects as appropriate.

Contract delivery requirements for each RAS program are specified in the following sections. The contract laboratory is required to deliver all analytical results and quality control (QC) data for each Sample Delivery Group (SDG) in one data package. An SDG is defined by one of the following, whichever occurs first:

- o each Case of field samples; or
- o each twenty field samples within a Case; or
- o each fourteen calendar day period during which field samples in a Case are received, beginning with the receipt of the first sample in the SDG.

Laboratories are subject to financial considerations for late delivery and incentives for early delivery of the data package. Illegible or incomplete data reports are unacceptable, and the laboratory must resubmit readable versions of any illegible pages.

The CLP QC program for RAS laboratory analysis is structured to provide consistent results of known and documented quality. Sample data packages contain QC documentation that allow an experienced chemist to determine the quality of the data and its applicability to each sampling activity. In addition, laboratory contracts contain provisions for sample reanalysis if specified QC criteria are not met by the contract laboratory. Each CLP laboratory is also encouraged to develop additional internal QA/QC procedures.

The minimum QC requirements of the RAS programs consist of both an initial and ongoing demonstration of laboratory capability to generate acceptable performance with the contract methods. The contract laboratory must demonstrate that instrument calibration criteria have been met, that interferences from the analytical system are under control, and that spike and duplicate recoveries falling outside contract acceptance windows are attributable to sample matrix interferences and not to laboratory analytical errors. The QC requirements for each RAS program are provided in the following sections.

## **A. Organic Routine Analytical Services**

### **1. Compounds Identified and Quantified**

The organic RAS program identifies and quantifies organic target compounds (VOA, B/N/A and pesticide/PCB fractions). These compounds are listed on the organic data reporting sheets in Appendix C.

In addition, the contract laboratory is required to execute a maximum of thirty NBS Mass Spectral Library searches for compounds not identified on the Target Compound List (TCL). The ten peaks of greatest apparent concentration in the VOA fraction and the twenty peaks in the B/N/A fraction are tentatively identified, and the concentration estimated, following a visual comparison of sample spectra with the nearest library matches. The tentative identification of non-target organic compounds provides information on potential organic contaminants outside of the analytical parameters of the RAS program.

### **2. Volumes Required and Preservation Techniques**

For low level organic water samples, a one gallon volume is required for extractables analysis; 80 mL is required for volatiles analysis. The extractables aliquot is collected in two 80-ounce, four 1-liter, or one 4-liter amber glass bottle(s). The volatiles aliquot is collected in two 40-mL glass vials. For medium level organic water samples, a four liter volume is required for extractables analysis; 80 mL is required for volatiles analysis. The extractables aliquot is collected in four 32-ounce glass jars; the volatiles aliquot is collected in two 40-mL glass vials. For low/medium level organic soil samples, a six ounce volume is required for extractables analysis and 240 mL is required for volatiles analysis. The extractables aliquot is collected in one 8-ounce glass jar, and the volatiles aliquot is collected in two 120-mL glass vials. Water and soil samples for volatile analysis should be collected so that the containers are completely filled, leaving no headspace. Because it is not certain whether a sample is actually low or medium level, samplers should collect volumes as specified for low level samples, but follow packaging and shipment procedures as prescribed for medium level samples. Packaging and shipment procedures are detailed in Chapter III, Section D.

Water samples for VOA analysis should be preserved with HCL. No chemical preservation is necessary for water samples for extractables analysis or for soil samples.

For a laboratory to perform matrix spikes, matrix spike duplicates, and contractual reanalyses, triple the sample volume is required for at least one sample in twenty of the same concentration and matrix for each Case. For water samples, one field blank should be supplied per Case, and one volatile trip blank should be supplied per shipment. No additional volume is required for duplicate analyses of soil samples. EMSL/LV supplies soil blanks to the Regions; aqueous blanks must not be used for soil samples. If the sampler does not provide sufficient volume, analysis of all required parameters and complete QA/QC determinations may not be possible. If this occurs, SMO will contact the RSCC to determine appropriate adjustments in analysis.

### 3. Contract Delivery Requirements

The organic RAS program specifies contractual requirements for sample extraction, volatile analysis and data reporting. These requirements include:

- o Completion of sample extraction for water samples within five days of sample receipt and for soil samples within ten days of sample receipt;
- o Completion of volatile analysis within ten days of sample receipt;
- o Completion of extractable analysis and reporting of data within thirty-five days of sample receipt.

Each organic RAS data package includes the following components:

- o Narrative report describing analytical problems encountered and internal QC processes applied.
- o Copies of sample Traffic Reports.
- o Quality control summary containing surrogate, method blank, matrix spike and matrix spike duplicate analyses recoveries, and instrument tuning and performance information.
- o Sample data including tabulated results of the organic target compounds identified and quantified, and the tentative identification and estimated concentration of up to thirty non-target organic compounds in greatest apparent concentration reported in ug/L or mg/kg.
- o Raw sample analytical data including sample chromatograms, spectra, quantitation reports and calculations.
- o Standards data package including chromatograms, spectra and data system printouts, and initial and continuing calibration reports.
- o Raw QC data package documenting instrument tuning and analytical QC criteria.

Each organic RAS package submitted to SMO must be accompanied by a diskette which contains machine readable information. This information must be sufficient to produce all data on the hard copy summary reporting forms. Explicit formats for diskette records are specified in the analytical Statement of Work. The organic RAS delivery requirements and copies of organic data reporting sheets are contained in Appendix C.

### 4. Analytical Protocols

The standardized organic analytical methods are based on Federal Register (FR) Methods 625 (B/N/A), 608 (pesticide) and 624 (VOA). Analysis for organic target compounds includes an optional GC screen (to determine appropriate dilution fraction or aliquot sizes for GC/MS analysis), GC/MS analysis (B/N/A and VOA) and GC/EC analysis (pesticide/PCB).

**a. Water and Soil Methods**

Water samples (VOA, B/N/A and pesticide/PCB fractions) are prepared and/or solvent extracted. Soil samples (B/N/A and pesticide/PCB fractions) are prepared by sonification prior to solvent extraction. Extracts are cleaned up using optional column chromatography techniques when necessary.

Organic target compounds are identified and quantified by GC/MS for VOA and B/N/A fractions and by GC/EC for the pesticide/PCB fraction. In addition, the twenty highest non-TCL B/N/A peaks and the ten highest non-TCL VOA peaks are tentatively identified and their concentrations estimated using a forward search of the NBS Mass Spectral Library.

**b. Contract Required Quantitation Limits**

Low level analysis contract required quantitation limits (CRQLs) for water samples are based on CRQLs for each organic compound using FR Methods 624, 625 and 608 and are at the part-per-billion (ppb) level. Approximate achievable sample quantitation levels for low water and low/medium soil samples can be calculated based on the sample size and on concentration/dilution factors.

CRQLs are provided for analytical guidance since the levels are highly matrix dependent. Matrix interferences vary considerably depending on the nature and homogeneity of the sample, on the interferent contaminants which coextract from the sample, and on the sample volume taken for analysis.

**5. Contract Quality Control Requirements**

QC procedures that must be performed and documented under the organic RAS program include, but are not limited to, the following:

Instrument QC procedure:

- o GC/MS instrument tunes for both volatile and semivolatile compound analyses.
- o Initial multilevel calibration for each target compound.
- o Continuing calibration for each target compound.

Sample QC procedure:

- o Addition of surrogate compounds to each sample and blank for determining percent recovery information.
- o Duplicate matrix spike analysis.
- o Method blank analysis.

Certain QC procedures demonstrate that the instrument is operating within contract specifications. These procedures include:

- o Demonstration that the two tuning compounds (DFTPP for extractables and BFB for volatiles) meet the defined ion abundance criteria.
- o Determination of an average response factor based on a calibration using five concentrations of each target compound. Specification of a subset of target compounds that must meet a defined relative standard deviation and minimum response factor.
- o A continuing calibration at a single concentration for each target compound where specified compounds are flagged as controls and must meet defined percent difference from the initial response factor or a new initial calibration must be performed.

Other QC procedures are required to demonstrate the quality of the analytical data generated. These procedures include:

- o Addition of surrogate spikes to all samples and blanks to monitor sample preparation and analysis and to provide percent recovery information for each sample so that the suitability of the method for each sample, regardless of matrix, may be established.
- o Analyses of duplicate matrix spiked samples to display the precision of the method for the particular matrix and also to provide percent recovery information for defined target compounds (specified matrix spikes) as for surrogates.
- o Analysis of reagent blanks for each Case or each set of twenty samples (whichever is less) and for each matrix within a Case to ensure that laboratory contaminants are not reflected in data results.

## **B. Inorganic Routine Analytical Services**

### **1. Analytes Identified and Quantified**

The inorganic RAS program identifies and quantifies metals and cyanide. These analytes are listed on the inorganic data reporting forms in Appendix C.

### **2. Volumes Required and Preservation Techniques**

For low level inorganic water samples, a one liter volume is required for metals analysis and a one liter volume is required for cyanide analysis. These samples should be collected in a 1-liter polyethylene bottle. For medium level inorganic water samples, a sixteen ounce volume is required for metals analysis and a sixteen ounce volume is required for cyanide analysis. These samples should be collected in a 16-ounce glass jar. For low/medium level soil samples, a six ounce sample volume is required for both metals and cyanide analyses. These samples should be collected in an 8-ounce glass jar.



Different preservation techniques apply to the metals and cyanide portions of low level water samples. For "total" metals analysis, the sample is acidified to  $\text{pH} < 2$  with  $\text{HNO}_3$ . ("Total" meaning inclusion of particulate and dissolved fractions.) For dissolved metals analysis, the sample is filtered and then acidified to  $\text{pH} < 2$  with  $\text{HNO}_3$  at the laboratory. If the sample contains a significant particulate fraction, acidification without filtration could result in deceptively high metal values for the water sample. Varying amounts of particulate matter can also give large differences in metal values for duplicate acidified water samples. The following guidelines should be utilized for the cyanide aliquot:

- o Test a drop of sample with potassium iodide-starch test paper (KI-starch paper). A resulting blue color indicates the presence of oxidizing agents and the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.6 g of ascorbic acid for each liter of sample volume.
- o Test a drop of sample on lead acetate paper moistened with acetic acid buffer solution. Darkening of the paper indicates the presence of  $\text{S}_2^{2-}$ . If  $\text{S}_2^{2-}$  is present, add powdered cadmium carbonate until a drop of the treated solution does not darken the lead acetate test paper. Filter the solution before raising the pH for stabilization.
- o Preserve samples with 2 mL of 10 N sodium hydroxide per liter of sample ( $\text{pH} > 12$ ).
- o Store the samples at  $4^\circ\text{C}$  until the time of analysis.

No chemical preservation is required for medium level water samples or for low/medium level soil samples unless otherwise directed.

For homogenization of water samples, the contract laboratory shakes the sample in its original sample container and transfers 100 mL aliquots to a 250 mL beaker. For water samples with a high solids content, the user can specify that the sample not be mixed and the analysis be performed on the supernatant. For homogenization of soil samples, the laboratory thoroughly mixes the contents of the sample container. For soil samples with significant amounts of water, the user has the option to specify that the supernatant be decanted and the remaining sample be mixed thoroughly and analyzed.

If it is not certain whether a sample should be categorized as low or medium concentration, volume should be collected and the sample preserved as specified for low level samples. Packaging and shipment procedures should be followed as designated for medium level samples. For water samples, one field blank should be supplied for each Case. Soil blanks are currently not available, and the user should not submit soil field blanks for analysis. If the user submits a rinsate blank with a Case of soil samples, the blank will be treated as a separate aqueous matrix sample with full QC, and accordingly, a sufficient volume for analysis should be provided to the laboratory. When a suitable soil blank material becomes available, EMSL/LV will supply one soil blank for each Case. No additional volume is required for duplicate analyses of water or soil samples; however, the user may specify that the duplicate and matrix spike be performed on a particular sample. If the sampler does not provide sufficient volume, analysis of all required parameters and complete QA/QC determinations may not be possible. If this occurs, SMO will contact the RSCC to determine appropriate adjustments in analysis.

### 3. Contract Delivery Requirements

The inorganic RAS program specifies the completion of metals and cyanide analysis and the submission of the final data package within thirty-five days following sample receipt at the laboratory. Each inorganic RAS data package includes the following components:

- o Cover page listing the samples included in the report and narrative comments describing problems encountered in analysis.
- o Tabulated results, reported in ug/L or mg/kg, of inorganic analytes identified and quantified. These results include a brief description of the sample. Individual analytical results are flagged by the laboratory when QC indicates potential bias due to matrix effects, homogeneity, etc.
- o QC results for preparation blanks, calibration blanks, calibration verification standards, matrix spikes, matrix spike duplicates, laboratory control samples, interference check samples, analytical spikes and serial dilution analyses.
- o Tabulation of instrument detection limits determined in pure water solutions.
- o Digestion/distillation logs, sample Traffic Reports, and raw data system printouts identifying calibration standards, calibration blanks, preparation blanks, samples and any atypical dilution, duplicates, spikes, interference checks and any instrument adjustments or apparent anomalies on the measurement record.

Each inorganic RAS package submitted to SMO must be accompanied by a diskette which contains machine readable information. This information must be sufficient to produce all data on the hard copy summary reporting forms. Explicit formats for diskette records are specified in the analytical Statement of Work. A summary of inorganic RAS delivery requirements and copies of data reporting forms are contained in Appendix C.

### 4. Analytical Protocols

The standardized inorganic analytical methods are based on FR Methods, EPA Methods for Chemical Analysis of Water and Wastes, and Test Methods for Evaluating Solid Waste (SW-846). Analysis for specified metals and cyanide is performed by flame, furnace and cold vapor atomic absorption (AA), colorimetric, distillation, and inductively coupled argon plasma (ICP) methods.

#### a. Water and Soil Methods

Samples for metals analysis are prepared and acid digested. The digestate is filtered to remove insoluble materials prior to analysis. Sample are analyzed by AA or ICP methods, and dilutions are performed where any analyte concentration exceeds the calibrated range.

A quantitative determination for cyanide is made by midi-distillation and colorimetric or titrimetric analysis. Mercury is quantitated in water samples by the cold vapor technique.

**b. Contract Required Quantitation Limits**

Inorganic RAS contracts contain minimum CRQLs that must be met by all laboratories for each of the metals and cyanide in pure water. On a quarterly basis, the contract laboratories are required to verify that their instrument detection limits (IDLs) meet the CRQLs.

CRQLs for low level water samples can be achieved in the ppb to low part-per-million (ppm) range; CRQLs for medium level water and low/medium level soil samples can be achieved in the low- to mid-ppm range. Matrix interferences and other sample parameters that vary with sample nature and homogeneity, with interferent contaminants that coextract from the sample, and with the analytical method can affect quantitation levels. Since achievable quantitation levels are dependent on the inorganic species and matrix of each sample, the laboratory must estimate levels based on extrapolations from the pure water IDLs. The laboratory brackets results below the CRQL to indicate a value near the IDL. Although data is reported down to the pure water IDL, results below the CRQL should be used with caution.

**5. Contract Quality Control Requirements**

Inorganics RAS contracts define extensive QA procedures that must be performed and documented. These include, but are not limited to, the following:

- o Initial calibration verification,
- o Continuing calibration verification,
- o ICP interference check sample analysis,
- o ICP serial dilution analysis,
- o Preparation blank analysis,
- o Spiked sample analysis,
- o Duplicate sample analysis,
- o Furnace AA QC analysis, and
- o Laboratory control sample analysis.

The instrument QC operations include initial and continuing calibration checks which are performed daily and/or every ten samples. These checks determine that the analytical system is meeting contract required criteria.

Analytical QC operations include:

- o ICP Interference Check Sample Analysis: Performed at least twice per eight hour shift to verify interelement and background correction factors.
- o ICP Serial Dilution Analysis: Performed for samples of a similar matrix and concentration for each Case of samples, or for each twenty samples received (whichever is more frequent), to ascertain whether significant chemical or physical interferences exist due to sample matrix.

- o **Preparation Blank Analysis:** Performed for each batch of samples or for each set of twenty samples to ascertain whether sample concentrations reflect contamination.
- o **Spiked Sample Analysis and Duplicate Sample Analysis:** Performed for each concentration and matrix within a Case of samples, or for each set of twenty samples of a similar matrix within a Case, to provide information concerning sample homogeneity, analytical precision and accuracy, and the effect of the sample matrix on the analytical methodology, and to enable the Agency to evaluate the longterm precision of the method.
- o **Furnace AA QC Analysis:** Required for quantitation; incorporates duplicate injections and analytical spikes in order to evaluate the precision and accuracy of the individual analytical determinations on each sample.
- o **Laboratory Control Sample Analysis:** Standards carried through sample preparation and analysis procedures to document the performance of the entire analytical process. On a quarterly basis laboratories verify their instrument detection limits, ICP linear ranges, ICP interelement correction factors and ICP integration times.

### **C. Dioxin Routine Analytical Services**

#### **1. Isomer Identified and Quantified**

The dioxin RAS contract method identifies and quantifies the 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) isomer. No concentration levels are designated in the dioxin program. All samples suspected to contain dioxin are considered hazardous and should be handled accordingly.

#### **2. Volumes Required**

The sample volume required for dioxin analysis is four ounces of soil/sediment or two liters of water. Each soil sample should be collected in either one 4-ounce glass jar or one 8-ounce glass jar. Each water sample should be collected in two 1-liter amber glass bottles. The collection of more than the required sample volume is strongly discouraged due to the hazardous nature and difficulty in disposing of dioxin-contaminated waste.

One or more field blanks should be included with each sample batch (no more than 24 samples). A rinsate sample, consisting of trichloroethylene used in rinsing sampling equipment, may be included in a batch. One sample with duplicate volume should be collected for duplicate analyses. QA samples, provided by EMSL/LV, should be included as part of the sample batch.

#### **3. Contract Delivery Requirements**

The dioxin RAS program specifies the completion of sample extraction, analysis and data reporting within twenty-one days of sample receipt at the laboratory. The delivery

requirements include automatic reextraction and reanalysis of samples when certain criteria are not met in the initial analysis. Each dioxin RAS data package includes the following components:

- o Completed data reporting sheets with appropriate selected ion current profiles (SICPs) and spectra attached indicating instrumental (GC/MS) operating parameters during data acquisition and including all rejected sample runs.
- o Results of analyses of multilevel concentration calibration solutions including SICPs, calculated response factors and computer-generated quantitation reports.
- o SICPs generated during each performance check solution analysis and each concentration calibration solution analysis.
- o Chronological list of all analyses performed.

A summary of dioxin RAS delivery requirements and copies of data reporting forms are contained in Appendix C.

#### **4. Analytical Protocols**

##### **a. Soil and Water Methods**

EPA-developed methods for the analysis of 2,3,7,8-TCDD are performed on a batch basis. A sample batch consists of up to twenty-four field samples and normally includes an equipment rinse solvent (trichloroethylene or hexane) sample, one or more field blanks, and a QA sample.

Prior to analysis, soil samples are prepared, homogenized and centrifuged when necessary. All samples are then solvent extracted according to matrix. The concentrated extract is analyzed by GC/MS using fused silica capillary column techniques. The 2,3,7,8-TCDD isomer is identified and quantified using selected ion monitoring (SIM) GC/MS instrumentation and data systems with the capability to acquire, store and retrieve SIM data for six ions.

##### **b. Contract Required Quantitation Limits**

The RAS contract method provides procedures for the detection and measurement of 2,3,7,8-TCDD in soil and water samples at concentrations as low as one ppb. Column chromatography and other clean up procedures are used to eliminate coextracted sample components, such as PCBs, which may interfere with the detection of very low levels of TCDD. Matrix interferences may also occur depending on the nature and homogeneity of the sample, and may prevent the achievement of the lowest CRQLs.

#### **5. Contract Quality Control Requirements**

Dioxin RAS contracts define extensive QC procedures that must be performed and documented. These include, but are not limited to, the following:

- o Initial and continuing calibration and instrument performance checks.
- o Reagent blank analysis.

- o Field blank analysis.
- o Fortified matrix spike analysis (2,3,7,8-TCDD spiked field blank).
- o Rinsate (equipment solvent) sample analysis.
- o Duplicate sample analysis.
- o Reanalysis including reextraction (and/or additional clean up of the sample extract) when QC criteria are not met in the initial analysis.

The instrument QC operations include initial and continuing calibration and instrument performance checks. Continued calibration is performed at the beginning of each twelve hour shift. Performance checks are performed at least twice during each twelve hour shift to demonstrate continued acceptable GC/MS resolution, sensitivity, response factor reproducibility, and mass range calibration, and to validate sample data.

Analytical QC operations include:

- o Reagent blank analysis to demonstrate that identified compound concentrations do not reflect laboratory contamination.
- o Field blank analysis to provide information on false-positive results, on the matrix effect of the sample on the analytical methodology, and on the accuracy of the method.
- o Rinsate sample analysis to ensure that samples have not been contaminated by sampling equipment.
- o Duplicate sample analysis to determine precision of the method.

#### **D. Special Analytical Services**

In addition to the standardized analyses available under the RAS program, the CLP provides Regional clients with limited specialized analyses under the SAS program. While these analytical services are beyond the scope of RAS contract protocols, they are consistent with CLP objectives. Services provided through the SAS program include fast turnaround analyses, verification analyses, analyses requiring lower detection limits than RAS methods provide, identification and quantification of nonpriority pollutant and non-TCL constituents, general waste characterizations, analysis of nonstandard matrices and other specific analyses.

As part of the SMO contract with EPA, Viar and Company solicits, awards and administers SAS subcontracts. By utilizing subcontracts, the CLP can procure specialized services in a timely manner on an as-needed basis. Due to the often unusual nature of SAS requests, users must plan their projects in advance to allow SMO sufficient time to procure these services.

For each SAS request, the client provides SMO with the necessary analytical methods and QA/QC requirements. SMO procures SAS by subcontracting with RAS laboratories with IFB contracts in the appropriate analytical program. When RAS laboratories cannot meet the analytical requirement of the SAS, requests are solicited to other laboratories which have demonstrated the ability to meet program performance requirements. RAS contract

laboratories are evaluated for current RAS performance before they are considered for SAS solicitations, and are not solicited for SAS work if deficient in this area. Other laboratories qualify to perform certain types of SAS work by successfully completing performance evaluation sample analyses or by justification of unique analytical capability.

Once the available laboratory community is determined, SMO contacts a minimum of five laboratories (contingent upon availability of a particular analytical service) and describes the requirements via telephone. Laboratories are asked to bid firm, fixed price(s) for the performance of specific types of analyses on a defined number of samples. SMO evaluates laboratory bids in terms of bid price and responsiveness to the specified task. The SAS is awarded to the lowest bidding laboratory which responds to the client's analytical requirement. A written, individual SAS subcontract agreement is then made between the laboratory and Viar and Company.

A laboratory's ability to bid for SAS work and the prices being bid may vary depending on the size or scope of the analytical request, data turnaround requirements and analytical parameters of a particular task, weekly RAS sample loading, and laboratory operating conditions at the time of solicitation. Due to the fluctuation of these factors on a weekly and often daily basis, the CLP may not be able to accommodate all SAS requests. SAS services are provided on a first-come basis; however, Agency requirements can necessitate that certain work be given priority. In this event, SMO notifies the involved RSCCs who determine Regional sampling priorities.

SAS requests are separated into two basic categories, "RAS Plus SAS" and "All SAS". These categories are utilized in defining client requests and pursuant SAS solicitation and contract award. Analytical services available through the SAS program are described below.

## 1. RAS Plus SAS

### a. Fast Turnaround

Fast turnaround requests require the application of existing RAS analytical parameters, methodologies and detection limits with a shorter timeframe for performance of analysis and/or delivery of data. Procurement for fast turnaround SAS is dependent upon program sample load, laboratory capacities and laboratory operating conditions at the time of the request. Because of constant fluctuations of these factors, it is not possible to obtain fast turnaround service on an unlimited basis. Fast turnaround contracts are solicited only in situations of demonstrated need and are used primarily to support EPA emergency actions and to meet impending litigation deadlines.

The following illustrates common "RAS Plus SAS" fast turnaround requests. The SAS portion is underlined:

- o RAS volatile organic target compound analysis with VOA analysis and data delivery in seven days.
- o RAS inorganic target compound analysis with data turnaround in fourteen days.
- o RAS dioxin target compound analysis with data turnaround in ten days.

**b. Special Requirements in Addition to RAS**

A client may need to access the standardized RAS programs and add to the contract requirements. The following examples illustrate common "RAS Plus SAS" requests. The SAS portion is underlined:

**(1) *Organic***

- o Volatile target compound analysis at lower detection limits than required by the IFB.
- o Full organics analysis with additional non-target pesticide/herbicide compounds.
- o Pesticide target compound analysis with minor alterations or additional procedures applied.
- o B/N/A target compound extraction with analysis by a non-RAS method.

**(2) *Inorganic***

- o Metals and cyanide analyses plus non-RAS parameters - nitrate, sulfate, ammonia, sulfide, total organic carbon and chloride.
- o Metals and cyanide analyses with special rigorous sample homogenization requirements.
- o Metals analysis at lower detection limits than required by the RAS requirements.
- o RAS metals and cyanide analysis with minor alterations or additional analytical procedures applied.

**(3) *Dioxin***

- o 2,3,7,8-TCDD analysis of soil/sediment samples with a detection limit lower than the one ppb required by the RAS contracts.
- o 2,3,7,8-TCDD analysis by the RAS protocol plus analysis of other dioxin or furan isomers\*.

**2. All SAS**

CLP clients frequently request types of analyses that are not directly applicable to the RAS program. These requests occur most often with samples of difficult or unusual matrices and measurements of analytical parameters not provided through the RAS program. Five categories of "All SAS" requests are described in the following sections.

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\*Future RAS protocol.



a. Organic

- o Seven TCL Aroclors analysis only (i.e., not the entire IFB pesticide fraction).
- o Non-target compound analyses\*.
- o Organic extraction of non-aqueous and non-soil/sediment samples (e.g., oil, tar or biological tissue samples by a non-RAS extraction procedure).
- o Organic analysis by non-RAS methods.

b. Inorganic

- o Specified RAS element analysis only (e.g., cadmium, mercury and selenium).
- o Non-RAS parameter analysis (e.g., total organic carbon, Sulfate, TSS, EP toxicity tests).
- o Any inorganic preparation/analysis of non-aqueous and non-soil/sediment samples (e.g., oil, tar or biological tissue).
- o Metals analysis by non-RAS methods.

c. Dioxin

- o 2,3,7,8-TCDD in fish tissue (e.g., matrix other than soil/sediment).
- o 2,3,7,8-TCDF (furan) in any matrix\*.
- o Total tetra- through octa- dioxin and/or furan classes in varied matrices\*.
- o Analysis by HRGC/HRMS or GC/MS/MS\*.

d. High Concentration Sample Analysis - Organic and Inorganic\*

- o Organic extraction and analysis for target compounds by GC/MS with tentative identification of thirty non-target compounds of greatest concentration.
- o Inorganic preparation/analysis for total metals including four procedures: KOH fusion, pneumatic nebulization ICP, hydride generation ICP, and mercury analysis. In addition to metals, cyanide and sulfide are quantitated.

e. Special Topics Analysis

The SAS program can usually accommodate unusual analytical requests on an "All SAS" basis when sufficient lead-time is allowed and complete methodology and QA/QC specifications accompany the request. These types of analyses include, but are not limited to, the following:

- o Biological samples (e.g., fish, turtle tissue) for specific organic, inorganic or dioxin analyses.
- o Air samples (e.g., tenax, charcoal and flurosil tubes) for specific organic analyses.

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\*Future RAS Protocol

- o Wipe samples for specific organic or inorganic analyses.
- o Methods comparison/evaluation studies.
- o Asbestos analysis.
- o Acid deposition parameters.
- o NonSuperfund analytical services of any type.

### **3. Contract Delivery and Quality Control Requirements**

SAS contracts require delivery schedules for sample extraction, analysis and data reporting, and require laboratory QC procedures and reporting of QC parameters as defined by the client requestor. Delivery and QC requirements as detailed in RAS program contracts may be used as a guide but must be specified by the client at the time of request. The requestor should specify all deliverables required to ensure that the appropriate data packages are received. Clients are encouraged to maintain a high level of QC in all analysis request, unless there is substantial reason for deleting certain QC requirements.

## **E. Analytical Methodology Improvement/Development**

### **1. Protocol Standardization and Improvement**

CLP participants are constantly refining and improving analytical protocols to maintain state-of-the-art status and to reflect newly defined or changed requirements of the Superfund effort. In order to accomplish this effort, all program participants submit comments or suggestions to the NPO on an ongoing basis. The NPO then reviews all submitted information and considers recommendations for program application on a periodic basis.

Since 1982, the NPO has utilized technical meetings as a means to consistently employ the scope of available resources in updating analytical program methodologies and data reporting requirements. Technical meetings are initiated by the NPO on a periodic basis and consist of workgroups, caucuses and an annual conference. Participants of these sessions include the Regions, the NPO, EMSL/LV, EMSL/Cincinnati, NEIC, SMO, contract laboratories, program support contractors, and other EPA programs and government agencies, as appropriate. These meetings are instrumental in improving CLP protocols and orienting deliverables to user needs.

EPA personnel review the discussions of the technical meetings and compile recommendations for protocol changes. Following NPO approval of recommended changes, existing laboratory contracts are modified by the Contracting Officer to include the recommended revisions. Whenever possible, all laboratory contracts within an analytical program are changed concurrently to maintain consistency within the program. NPO-approved protocol revisions are included in any new IFB solicitations.

**2. Method Development**

Development of new analytical methods may be initiated by a newly identified or redefined Agency analysis requirement. Analytical methods utilized in the CLP are based on methodologies developed and approved by EPA. The NPO, EMSL/LV, EMSL/Cincinnati, the Regions and the contractor community have historically contributed to the development of new program analytical methodologies. Methods are reviewed by several sources and are tested prior to implementation to the extent possible to meet program requirements.

## **CHAPTER III**

### **UTILIZATION OF ANALYTICAL SERVICES**

The CLP provides clients with prompt access to laboratory services through a documented system of sample scheduling. The CLP scheduling process is based on two fundamental requirements: 1) maintenance of ongoing communication among the RSCC, field sampler, SMO and laboratory personnel, and 2) correct use of sample scheduling and tracking documents by the RSCC, field sampler and laboratory personnel.

SMO coordinates the scheduling of sample analyses through the CLP and tracks the progress of samples from collection through final data production. To effectively match the analytical needs of program clients with the capabilities of contract laboratories, SMO documents current utilization, availability of resources and laboratory performance limitations for each program.

SMO is authorized to accept analytical requests only through the RSCC, which is established by the EPA Regional Administrator and is centered in each Region's Environmental Services Division or Waste Management Division. The RSCC, consisting of one or more identified individuals (primary and secondary), routinely places analytical requests with SMO and coordinates those requests throughout sample shipment and analysis. In addition, the RSCC is responsible for ensuring Regional compliance with the CLP's projection/allocation system. The primary RSCC determines analytical priorities for the Region when conflicts occur. Individuals interested in obtaining CLP analytical support should contact their Regional EPA office's RSCC (see Appendix B).

#### **A. Analysis Request Procedures**

##### **1. RAS Initiation Process**

###### **a. User Information Required**

To initiate a RAS request, the RSCC contacts the appropriate SMO Coordinator by telephone and provides a complete description of the analytical requirement. (SMO personnel are identified in Appendix B.) SMO requires the following information to initiate a RAS request:

- o Name of the individual RSCC.
- o Name(s), association and telephone number(s) of sampling personnel.
- o Name, city and state of the site to be sampled.
- o Superfund site/spill ID (2 digit alpha-numeric code).
- o Number and matrix of samples to be collected.
- o Type of analyses required.

Organic: Full (VOA, B/N/A and pesticide/PCB), VOA and/or B/N/A and/or pesticide/PCB, or VOA only fractional analyses.

Inorganic: metals and/or cyanide.

Dioxin: 2,3,7,8-TCDD.

- o Scheduled sample collection and shipment dates.

- o Nature of sampling event.
  - Preliminary Assessment
  - Site Investigation
  - Expended Site Investigation
  - Remedial Investigation/Feasibility Study
  - Remedial Design
  - Remedial Action
  - Enforcement Lead
  - Emergency Response (Removal)
  - National Priorities List Delete
  - Operation and Maintenance
  - State Lead Preliminary Assessment
  - State Lead Site Investigation
  - State
  - National Dioxin Study
  - Facility Assessment
  - Compliance Monitoring Effort
  - Enforcement
  - Ground Water Monitoring Task Force
  - Resource Conservation and Recovery Act
  - Office of Water
  - Clean Air
- o Suspected contaminants associated with the sample and/or site.
- o Other pertinent information which may affect sample scheduling or shipment (i.e., anticipated delays due to site access, weather conditions, sampling equipment).
- o Name(s) of Regional or contractor contacts for immediate problem resolution.

The RSCC is responsible for estimating the number and types of samples and the sample shipment dates for the analytical request. Overestimation of the number of samples to be collected or miscalculation of shipment dates unnecessarily ties up available laboratory capacity, and thus renders the program less than maximally responsive to all clients. Underestimation of the number and types of samples to be collected may result in unavailable services for any additional analyses needed.

b. Lead-time Requirement

At least one week prior to the scheduled start of a planned sampling activity, the RSCC telephones SMO to place a specific request for RAS services. The RSCC is required to provide scheduling information to SMO by noon on the Wednesday of the week prior to sample shipment. This lead-time facilitates laboratory scheduling and resolution of questions concerning sampling and analysis procedures, and allows the sampler adequate time to prepare the required sample documentation. Advance scheduling is available and should be utilized whenever possible.

c. Case Number Assignment and Laboratory Scheduling

At the time of request, SMO assigns a sequential Case number to each RAS sampling activity for identification throughout sample tracking and data production. A Case number designates a single group of samples collected at one site or geographical location during a predetermined and finite time period. The RSCC records the Case number and uses it in referencing that request throughout sampling and analysis.

SMO then schedules the requested analyses through an appropriate RAS laboratory. Laboratory selection is determined by the types of analyses, number of samples, contract capacity, sample balance among the various laboratories, and laboratory loading and instrument conditions. Organic laboratory selection is also based on the Regional Distribution of Laboratories System developed by the NPO and designed to minimize the number of laboratories producing data for any one Region. When possible, the nearest available laboratory is assigned in order to minimize sample shipping costs.

Once RAS laboratory assignments are made, SMO contacts the RSCC to confirm the field investigation plans, identify the laboratories to be used for the Case, and answer any further questions regarding program procedures or documentation. At that point, the RSCC must indicate all known or anticipated sample scheduling changes. Any other changes occurring after this time should be communicated to SMO immediately upon identification to ensure the timely resolution of conflicts and the optimal allocation of program resources. After the initial placement of the RAS request, the RSCC may choose to assign a logistical contact, such as the team leader in the sampling effort, to coordinate with SMO in finalizing sampling requirements, and initiating and arranging sample shipment.

d. User Knowledge of Analytical Protocol

Each RSCC is responsible for acquiring and maintaining a working knowledge of current RAS protocols and analytical services. SMO provides each Regional DPO (listed in Appendix B) with Master Copy notebooks of each RAS program IFB Statement of Work (SOW). The Master Copy notebooks are periodically updated to reflect program protocol changes.

The SOW represents the standardized requirements which each individual RAS laboratory is contractually bound to follow. The analytical SOWs contain specific information on sample types suited to RAS analysis, compounds identified and quantified, analytical methods, protocols, detection limits, deliverable requirements, and quality control requirements. Program users should consult the appropriate SOW to confirm that the RAS program is suited to an analytical request.

## **2. SAS Initiation Process**

a. User Information Required

Analytical requirements differing from RAS parameters are processed through the SAS program as described in Chapter II, Section D. To initiate a SAS request, the RSCC contacts

the appropriate SMO Coordinator by telephone and provides a complete description of the analytical requirement. SMO requires the following information to initiate a SAS request:

- o Name of RSCC.
- o Name(s), association and telephone number(s) of sampling personnel.
- o Name, city and state of the site to be sampled.
- o Superfund site/spill ID (2 digit alpha-numeric code).
- o Number and matrix of samples to be collected.
- o Specific analyses required, appropriate protocols and QA/QC.
- o Required detection limits.
- o Matrix spike, matrix spike duplicate, duplicate or LCS frequency, if applicable.
- o Data turnaround and data format.
- o Justification for fast turnaround request, if appropriate.
- o Scheduled sample collection and shipment dates.
- o Nature of sampling event.

Preliminary Assessment

Site Investigation

Expended Site Investigation

Remedial Investigation/Feasibility Study

Remedial Design

Remedial Action

Enforcement Lead

Emergency Response (Removal)

National Priorities List Delete

Operation and Maintenance

State Lead Preliminary Assessment

State Lead Site Investigation

State

National Dioxin Study

Facility Assessment

Compliance Monitoring Effort

Enforcement

Ground Water Monitoring Task Force

Resource Conservation and Recovery Act

Office of Water

Clean Air

- o Suspected contaminants associated with the samples and/or site.



- o Other pertinent information which may affect sample scheduling or shipment (i.e., anticipated delays due to site access, weather condition, sampling equipment).
- o Name(s) of Regional or contractor contacts for immediate problem resolution.

In follow up to the verbal request, the RSCC must submit a completed SAS Client Request form to SMO. This form serves as the written record to clarify and confirm the client's requirement for specialized analytical work. A copy of the SAS Client Request form is included in Appendix D.

The RSCC is responsible for estimating the number and types of samples and the sample shipment dates for the SAS request. Overestimation of the number of samples to be collected or miscalculation of shipment dates unnecessarily ties up available laboratory capacity, and thus renders the program less than maximally responsive to all clients. Underestimation of the number and types of samples to be collected may result in unavailable services for any additional analyses needed. Depending on the size and extent of the miscalculation, the entire request may have to be resolicited and sampling plans postponed accordingly.

b. Lead-time Requirements

When a sampling activity has been planned, the RSCC telephones SMO and places the specific request for SAS services. Because SAS services are individually procured on a competitive basis, a minimum lead-time of two weeks is required to process a completely defined SAS request. More lead-time is strongly recommended whenever possible. SAS solicitation will not be started until the SAS requirements have been completely defined by the RSCC. Modifications to any SAS request will cause the entire process to begin again. Fully defined requests initiated with less than two weeks lead-time may not be solicited and awarded in time to meet the original shipment date.

Certain types of SAS requests require a longer lead-time. A minimum lead-time of two to three weeks is required for SAS requests which involve distribution of protocols (see item d, below). A minimum lead-time of four or more weeks is recommended for large scale, analytically complex or nonSuperfund SAS requests. Award of nonSuperfund SAS subcontracts may only be made after the appropriate funding process is complete. The RSCC should contact SMO several weeks in advance if there is a question regarding the lead-time needed to schedule a particular SAS request.

c. SAS Number Assignment and Laboratory Scheduling

At the time of request, SMO assigns a sequential SAS number to each SAS sampling activity for identification throughout sample tracking and data production. If SAS services are being provided in association with RAS services, SMO also assigns a Case number. Like the Case identification, the SAS number designates a single group of samples collected at one site or geographical location during a predetermined and finite time period. The RSCC records the SAS number and Case number (if applicable) and uses both numbers in referencing the request throughout sampling and analysis.

SAS laboratory selection is based on a verbal and written solicitation process for each individual request. This solicitation results in a written SAS award to the lowest qualified bidder. Once SAS laboratory assignments are made, SMO notifies the RSCC of the laboratories that will be performing the analyses.

The nature of the SAS laboratory solicitation process requires the RSCC to be as exact as possible with all elements of a request at the time of request. SMO understands that actual site conditions can vary considerably from expected conditions and necessitate changes in the sampling plan. However, the RSCC is responsible for notifying SMO immediately of any changes to allow sufficient time to amend the SAS contract(s) to meet the changed needs. If an original request is changed significantly, the original SAS contract will be voided, and the entire analysis effort will be resolicited. SAS resolicitation requires additional time before sample shipment can take place.

d. User Provided Analytical Protocol

At the time of request, the RSCC must provide the analytical methodology and quality control requirements to be utilized for the SAS request before SMO can initiate a solicitation. For SAS requests that are based on the use of amended RAS protocols, the RSCC must specify modifications or additions to these protocols. If such changes are extensive, the RSCC must submit changes under the SAS to SMO in written form two to three weeks in advance of scheduled sample shipment. For SAS requests which require use of a method that is not commonly available, the RSCC must submit the method two to three weeks in advance of sample shipment. Additional lead-time is required for protocol distribution and review by solicited laboratories.

SAS requests which cite the application of well known analytical publications do not require additional lead-time for distribution since laboratories have immediate access to this information. Examples of frequently utilized method manuals are as follows:

- o Methods for Chemical Analysis of Water and Waste, USEPA, 1983.
- o Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, USEPA Office of Water and Waste Management, 1983.
- o Standard Methods for the Examination of Water and Waste Water, APHA, AWWA, WPCF, Current Edition.

Further analytical references are supplied in Appendix F. The RSCC should contact SMO several weeks in advance if there is a question as to whether a particular method will require additional lead-time for distribution.

### 3. Procedures for Making Changes to Analytical Requests

The RSCC or designated logistical contact must immediately notify the appropriate SMO Coordinator of all changes in sampling plans before and during the sampling event and after shipment of samples to the laboratory. Changes in plans include changes in sample matrices, numbers of samples, analyses requested, detection limits, shipping dates, postponements or

cancellations. Failure to notify SMO of such changes can result in delay in sampling to accommodate scheduling changes, delay in start of analysis due to conflicts, unsuitability of a particular sample to an analytical program, or analysis data inappropriate for client purposes.

### **B. Regional Organic/Inorganic Allocation System**

The NPO has established an allocation system to equitably apportion available laboratory capacity to the Regions during periods of heavy sampling activity. Currently, capacity is available for the projected sample demand; however, when the allocation system is in effect, all organic and inorganic RAS and "RAS Plus SAS" Cases will be scheduled accordingly.

During the last month of each fiscal year quarter, the NPO provides the RSCC with the Region's monthly allocation of organic and inorganic sample analyses for the following quarter. The RSCC is responsible for planning monthly sampling activities in accordance with the NPO allocation.

Under the scheduling/allocation system, the RSCC requests sample analyses for all planned Regional sampling activities for a given week on the Wednesday preceding that week and assigns a priority, if requested by SMO, to each request. Upon receiving the Region's analytical requests, SMO makes laboratory assignments for the week and schedules received requests up to each Region's allocation limit. Requests in excess of the monthly allocations will not be processed by SMO until all Regional requests which fall within allocations have been placed at a laboratory. At this time, any excess laboratory capacity for the week is determined, and the NPO prioritizes Regional sampling requests that exceed allocations. SMO, utilizing available laboratory capacity, then makes laboratory assignments for sampling activities as prioritized by the NPO. For additional information concerning the allocation system, user's should contact SMO's Group Leader for Analytical Services (see Appendix B).

### **C. Sample Documentation**

Each sample processed by the CLP must be properly documented to ensure timely, correct and complete analysis for all parameters requested, and most importantly, to support the use of sample data in potential enforcement actions. The CLP documentation system provides the means to individually identify, track and monitor each sample from the point of collection through final data reporting. As used herein, a sample is defined as a representative specimen collected at a specific location of a waste site at a particular point in time for a specific analysis. A sample may reference field samples, duplicates, replicates, splits, spikes or blanks that are shipped from the field to a laboratory. Whenever questions arise, samplers should contact SMO for direction and clarification concerning the proper completion and distribution of CLP paperwork.

#### **1. Sample Traffic Report**

RAS organic and inorganic samples are documented with corresponding CLP sample Traffic Reports (TRs), a four part carbonless form. Each TR may document up to twenty samples

shipped to one CLP laboratory under one Case Number and one RAS analytical program. Samplers must complete the appropriate TRs for every shipment of RAS samples to a CLP laboratory. Copies of the two types of TRs, as well as examples of properly completed TR forms, are included in Appendix D.

TR forms must also be used when an individual sample is to be analyzed for both RAS and SAS parameters. A SAS Packing List is not required and should not be used in addition to the TR. Both the Case number and the SAS number must be entered at the top right of the form in order to clearly identify and track the sampling event. Samplers must take caution not to include the Case number on "All SAS" samples taken at the same site. Additionally, the sampler must briefly describe the SAS requirement on each TR (e.g., "VOA - 1 ppb detection limit").

Samplers record every sample on the TR form by completing the columns for sample number, sample description, concentration, RAS analytical fraction, special handling and station location. In addition, samplers complete the boxes for type of activity, site name, Regional information, analysis laboratory, sampling date and shipping information.

After completing the TR, the sampler includes the bottom two copies with the sample shipment to the laboratory, returns the top copy to SMO, and retains the remaining copy for their file. Upon receipt of the sample shipment, the laboratory documents sample condition and signs the TR. The laboratory returns a copy of the signed TR to SMO and retains a copy for their file. Copies of the signed TRs are provided to the RSCC as part of the data package.

SMO provides TR forms to each Region through the RSCC. The RSCC should contact SMO two or more weeks in advance to order additional TR forms.

## **2. Dioxin Shipment Record**

The CLP Dioxin Shipment Record (DSR), a four part carbonless form, is used as sample documentation for the RAS dioxin program. These forms must also be used for any "RAS plus SAS" dioxin samples. The DSR provides a record for one shipment batch of dioxin samples (up to twenty-four samples). A copy of the DSR, as well as an example of a properly completed DSR form, is included in Appendix D.

To provide a permanent record of each sample collected, the sampler records the appropriate Case number and batch/shipment number on the DSR form. The sampler then enters header information including type of activity, Regional information, shipping information and analysis laboratory. The sampler records sample matrix and description (e.g., soil/sediment field sample, solvent rinsate) for each sample by checking the appropriate box following each sample number.

After completing the DSR, the sampler includes the bottom two copies with the sample shipment to the laboratory, returns the top copy to SMO, and retains the remaining copy for their file. Upon receipt of the sample shipment, the laboratory documents sample condition and signs the DSR. The laboratory returns a copy to SMO and retains a copy for their file. Copies of the laboratory-signed DSRs are provided to the RSCC as part of the data package.

SMO provides DSR forms to each Region through the RSCC. The RSCC should contact SMO two or more weeks in advance to order additional DSR forms.

### **3. SAS Packing List**

For "All SAS" samples, samplers are to utilize the SAS Packing List (PL), a four part carbonless form. The PL provides space to list up to twenty samples on one form. SAS samples are numbered using the SAS number followed by a hyphen and progressive numerical designation, starting with 1 (e.g., 2000E-1, 2000E-2, 2000E-3, etc.). If the sampling activity extends over several days and more than one PL is used, care must be taken not to repeat sample numbers. A copy of the SAS PL, as well as an example of a properly completed PL form, is included in Appendix D. Regions should consult SMO to verify that the PL is appropriate to use in their situation.

The sampler completes the PL by recording the SAS number, site name and location, sampling date, shipment date, analysis laboratory, sampling office, sampler name and telephone number, individual SAS sample numbers, sample description and analytical parameters requested. After completing the PL, the sampler includes the bottom two copies with the sample shipment to the analysis laboratory. Following sample shipment, the sampler sends the top copy to SMO and retains the second copy as a file copy. Upon receipt of samples, the analysis laboratory documents sample condition and signs the PL, returns a copy to SMO and keeps a laboratory file copy. Copies of the laboratory-signed PLs are provided to the RSCC as part of the SAS data package.

SMO provides SAS PL forms to each Region through the RSCC. The RSCC should contact SMO two or more weeks in advance to order additional SAS PL forms.

### **4. Sample Number**

A unique sample number, recorded on the TR, DSR and SAS PL, identifies each sample. Inorganic and organic/VOA sample numbers have different formats and are not interchangeable. Strips of adhesive labels preprinted with individual sample numbers are provided by SMO with TR and DSR forms. Samplers must provide sample labels, marked in indelible ink with the appropriate SAS sample numbers, for use with "All SAS" samples.

The sampler affixes the sample label to the corresponding containers that make up the sample and, if appropriate, to the outside of the metal can in which the sample is packed (see Section D for packaging requirements). The top edge of the label should be placed at the level of initial sample volume so that any loss of volume can be easily detected. In order to protect the labels from the effects of water and solvent, labels are covered with clear, waterproof tape.

### **5. Sample Tag**

Each sample removed from a waste site and transferred to a laboratory for analysis is identified by a sample tag which contains specific sample information as defined by NEIC.

Sample tags are retained by the laboratory as physical evidence of sample receipt and analysis. Sample tags may be obtained through the Regional office; in some instances, sampling contractors may be required to provide their own sample tags.

The information recorded on the sample tag includes:

- o CLP Case/SAS No(s). - The unique number(s) assigned by SMO to identify the sampling event. (Entered under "Remarks" heading.)
- o CLP Sample No. - The unique sample identification number (from the TR, DSR, or PL) used to document that sample. (Entered under "Remarks" heading.)
- o Project Code - The number assigned by EPA to the sampling project.
- o Station No. - A two digit number assigned by the sampling team coordinator.
- o Date - A six digit number indicating the month, day and year of collection.
- o Time - A four digit number indicating the military time of collection.
- o Station Location - The sampling station description as specified in the project plan.
- o Samplers - Signatures of samplers on the project team.
- o Remarks - Case/SAS and sample numbers, as well as any pertinent comments, are entered here.
- o Tag No. - A unique serial number preprinted or stamped on the tag.
- o Lab Sample No. - Reserved for laboratory use.

Additionally, the sample tag contains appropriate spaces for noting that the sample has been preserved and indicating the analytical parameter(s) for which the sample will be analyzed. After the sample tag is completed, each tag is securely attached to the sample container. Samples are then shipped under chain-of-custody procedures as described in the following section. An example of a properly completed sample tag is provided in Appendix D.

## **6. Chain-of-Custody Record**

In accordance with Agency enforcement requirements, official custody of samples must be maintained and documented from the time of collection until the time of introduction as evidence during litigation. The following custody documentation procedure was developed by NEIC and is used in conjunction with CLP documentation (i.e., TR, DSR and SAS PL) for all samples processed through the program.

A sample is considered to be in an individual's custody if any of the following criteria are met: 1) the sample is in your possession or it is in your view after being in your possession; 2) it was in your possession and then locked up or sealed to prevent tampering; or 3) it is in a secured area. The team member performing the sampling is responsible for the care and custody of the collected samples until they are dispatched properly. In follow up, the sampling team leader reviews all field activities to confirm that proper custody procedures were followed during the field work.

The Chain-of-Custody Record is employed as physical evidence of sample custody. The sampler completes a Chain-of-Custody Record to accompany each cooler shipped from the field to the laboratory. Chain-of-Custody Record forms can be obtained through the Regional office.

The sampler records the project number, samplers' signatures and the Case and/or SAS number as header information on the Chain-of-Custody Record. The commonly known name of the site should not be included since CLP laboratories may perform work for the responsible party of that site. For each station number, the sampler indicates date, time, whether the sample is a composite or grab, station location, number of containers, analytical parameters, CLP sample number(s) and sample tag number(s). When shipping the samples, the sampler signs the bottom of the form and enters the date and time the samples are relinquished. The sampler enters shipper name and airbill number under the "Remarks" section on the bottom right of the form. A copy of the Chain-of-Custody Record, as well as an example of a properly completed custody record, is included in Appendix D.

The custody record is completed using waterproof ink. Any corrections are made by drawing a line through and initialing the error, then entering the correct information. Erasures are not permissible.

The original signature copy of the Chain-of-Custody Record is enclosed in plastic (with CLP sample documentation) and secured to the inside of the cooler lid. A copy of the custody record is retained for the sampler's files. Whenever samples are split with a source or government agency, a separate Chain-of-Custody Record should be prepared for those samples to indicate with whom the samples are being split and sample tag serial numbers from splits.

Shipping coolers are secured and custody seals are placed across cooler openings. As long as custody forms are sealed inside the sample cooler and custody seals remain intact, commercial carriers are not required to sign off on the custody form.

The laboratory representative who accepts the incoming sample shipment signs and dates the Chain-of-Custody Record to acknowledge receipt of the samples. Once the sample transfer process is complete, the laboratory is responsible for maintaining internal logbooks and records that provide a custody record throughout sample preparation and analysis.

## **D. Sample Packaging and Shipment**

### **1. Packaging Requirements**

Samples processed through the CLP must be packaged for shipment in compliance with current U.S. Department of Transportation and commercial carrier regulations. All required government and commercial carrier shipping papers must be filled out and shipment classifications made according to these regulations. (Consult Appendix F for shipping references.)

Waterproof, metal or hard plastic ice chests or coolers are the only acceptable type of sample shipping container. Inside the cooler, sample containers must be enclosed in clear plastic bags

so that sample tags and labels are visible. Water and soil samples suspected to be of medium/high concentration or soil samples suspected to contain dioxin must be enclosed in a metal can with a clipped or sealable lid (e.g., paint cans). The outer metal can must be labeled with the number of the sample contained inside. Containers which do not fit into paint cans should be double bagged.

Shipping containers should be packed with noncombustible, absorbent packing material (e.g., vermiculite) surrounding the sample bottles or metal cans containing samples to avoid breakage during transport. Earth or ice should never be used to pack samples; earth is a contaminant, and ice melts resulting in container breakage.

Water samples for low/medium level organic analysis and low/medium level cyanide analysis must be cooled to 4°C with ice when shipped. Shipping with ice is optional for soil samples for low/medium level organic analysis or low/medium level cyanide analysis. Ice is not required in shipping high concentration water or soil samples for organic analysis or for any matrix/concentration samples for metals or dioxin analysis. Ice should be in sealed plastic bags to prevent melting ice from soaking packing material which, when soaked, makes handling of samples difficult in the lab.

Low level inorganic and VOA water samples require chemical preservation. Users should consult Chapter II for preservation techniques.

TRs, DSRs, SAS PLs, Chain-of-Custody Records, and any other sample documentation accompanying the shipment must be enclosed in a waterproof plastic bag and taped to the underside of the cooler lid. Coolers must be sealed with custody seals in such a manner that the custody seal would be broken if the cooler were opened.

Shipping coolers must have clearly visible return address labels on the outside. Shipping coolers that are labeled in this manner will be returned to the sampler by the laboratory within fourteen days following laboratory sample receipt. A summary of correct sample packaging is illustrated in Appendix D.

## **2. Shipping Instructions**

All samples should be shipped through a reliable commercial carrier, such as Federal Express, Emery, Purolator or equivalent. Sampling offices are responsible for sample shipping charges.

Samples for organic analysis must be shipped for overnight delivery. If shipment requires more than a 24-hour period, sample holding times can be exceeded compromising the integrity of the sample analysis. Samples for inorganic analysis should be held until sampling for the Case is complete and then shipped for two-day delivery. In the RAS inorganic program, three days is the recommended period for collection of a Case of samples.

The NEIC/Denver and the ERT/Cincinnati hazardous waste site manuals provide extensive information on EPA-approved sample packaging and shipment techniques. References for these materials are provided in Appendix F. In addition, general questions concerning sample packaging and shipment may be directed to SMO.



### 3. Shipment Coordination

To enable SMO to track the shipment of samples from the field to the laboratory and ensure timely laboratory receipt of samples, the sampler must notify SMO of all sample shipments on the day of shipment. At that time, the sampler should provide the following information:

- o Sampler name and phone number.
- o Case number and/or SAS number of the project.
- o Site name/code.
- o Batch numbers (dioxin only).
- o Exact number(s), matrix(ces) and concentration(s) of samples shipped.
- o Laboratory(ies) to which samples were shipped.
- o Carrier name and airbill number(s) for the shipment.
- o Method of shipment (e.g., overnight, two-day).
- o Date of shipment.
- o Suspected contaminants associated with the samples or site.
- o Any irregularities or anticipated problems with the samples, including special handling instructions, or deviations from established sampling procedures.
- o Status of the sampling project (e.g., final shipment, update of future shipping schedule).

Sample shipments made after 5:00 p.m. EST should be called in to SMO at the start of business the next day (8:00 a.m. EST). SMO must be notified by 3:00 p.m. EST Friday of sample shipments intended for Saturday delivery. CLP laboratories remain open to receive Saturday shipments only upon advance notification by SMO and only when shipment information has been provided to SMO by the sampler.

The success of sample shipment coordination depends on the proper use and handling of the sample tracking forms and timely, complete communication among the RSCC, samplers, SMO and laboratories. Any postponements, cancellations, changes in the number or type of samples to be collected or changes in shipping dates must be communicated to SMO immediately. Appendix D contains a checklist for coordinating sample shipment.

## E. Procedures for Problem Resolution

### 1. Resolving Problems Concerning Sample Shipment and Analysis

Program laboratories routinely notify SMO upon encountering problems with sample receipt or during sample analysis. (Examples of these types of problems are listed in Appendix D.) In response, SMO immediately contacts the RSCC to relay the problem and to assist in formulating a solution. SMO then contacts the laboratory involved to communicate the

recommended action and to authorize processing of the sample(s) in question. Timeliness is the key to this type of problem resolution since delays could affect contractual time requirements for sample extraction and analysis and, if extreme, could invalidate the analysis.

Users should refer general questions regarding sample shipment, required sample analysis, laboratory contracts or the status of data deliverables on a particular Case or SAS to the appropriate SMO personnel. Technical questions regarding contract analytical procedures should be referred to the PO or the DPO of the laboratory through the NPO.

## **2. Resolving Problems Concerning Analytical Data**

In the CLP's Regional/Laboratory Communication System, authorized Regional personnel can contact specified laboratory personnel to resolve questions regarding the final data package. This system may only be used after laboratory data submission and may never be used to initiate additional analytical work to resolve data questions. All communications between laboratories and Regional contacts are recorded by each party on a Telephone Record Log. Documented information includes Case and/or SAS number, individuals making contact, subject of the discussion and its resolution. In follow up, the Region and laboratory send copies of completed telephone logs to SMO where the logs become a permanent part of the Case/SAS file. An example of the Telephone Record Log is included in Appendix D. Telephone Record Logs are available from SMO.

Prior to the laboratory's submission of the final data package, client queries regarding those analyses or data are handled through SMO. Depending on the nature of the question, SMO will respond or will direct the client to the appropriate NPO official for resolution. Comments regarding laboratory performance, whether positive or negative, should be directed in writing to the DPO of the laboratory with a copy provided to the PO.

## **CHAPTER IV**

### **AUXILIARY SUPPORT SERVICES**

The CLP provides several supplementary services that have developed as a natural adjunct to the program's analytical services. A description of each auxiliary service and the procedures for accessing the service are provided in the following sections.

### **A. Sample Bottle Repository Program**

#### **1. Types of Containers Available**

The Sample Bottle Repository program provides CLP clients with eleven types of cleaned, quality controlled sample containers for use in hazardous waste sampling collection. Sample coolers and sample preserving agents are not supplied through the program.

To ensure that no contamination exists that might affect sample data results, each container type is cleaned and quality control tested by specified procedures. These methods are directly related to the analyses that may be performed on samples collected in the container. The following chart lists the types of containers provided through the program and the type(s) of samples appropriate for collection in each container. To ensure appropriate quality control, samplers should use containers only to collect samples as listed on the following chart.

#### **2. Ordering Procedures**

The Sample Bottle Repository program may be accessed by Regional and contractor clients for sample collection under the Superfund program and other nonSuperfund Agency programs. Two individuals from each organization are designated as Authorized Requestors (ARs); only these individuals may place container requests through the program. Users interested in accessing the Repository program should contact their RSCC or SMO for further information. State personnel should access the program through their Regional EPA office.

Once a user has become authorized to request containers from a Repository, SMO provides them with a supply of Delivery Requests (DRs), a three part carbonless form, so that the AR can request containers directly from the Repository. Since the Repository can respond only to requests submitted by a designated AR, users must promptly notify SMO of any changes in AR designations.

Container requests are defined by the amount of time between the date the Repository receives the request (verbal or written) and the required delivery date:

- o Routine Request: Fifteen or more working days lead-time for delivery.
- o Fast Turnaround Request: More than three but less than fifteen working days lead-time for delivery.
- o Emergency Request: Less than three working days lead-time for delivery.

All DRs must be signed by an AR. For routine requests, the original copy of the completed DR is sent to the Repository at the address indicated on the form, the second copy is retained for the user's file, and the third copy is sent to SMO. Because of short lead-times, fast-

turnaround and emergency requests should be telephoned to the Repository at the number provided on the form. The written DR must be distributed per routine procedure to confirm the request.

Whenever possible, users should submit requests a minimum of two weeks in advance of the required delivery date to ensure timely and complete delivery of containers. The Repository may not be able to respond to all emergency and fast-turnaround requests; response depends on Repository inventory and in-house requests.

In the event that requested containers are no longer needed, the user must immediately contact the Repository to verbally cancel the request, follow up with a cancellation memorandum to the Repository, and send a copy of the memorandum to SMO. Cancellation memos, as well as all other project-related correspondence, should cite the appropriate DR number.

### **3. Shipment Information**

Upon receipt of the DR, Repository personnel begin preparing the request and schedule shipment. Repository personnel immediately notify the AR, if for any reason, the request cannot be met in full by the required delivery date. Often partial shipments can be arranged over several days to meet the AR's requirement. If concurrent requests are received at the Repository that cannot be filled in a timely manner and if partial shipments cannot be satisfactorily arranged, the Repository immediately notifies SMO. SMO then coordinates with the involved RSCC in determining the priority of container requests based on the Region's sampling needs.

Each carton in a shipment is marked "Box \_\_\_\_ of \_\_\_\_," and a Repository Packing List (PL) is included in Box 1 of each shipment so that the recipient can verify that the entire shipment has been received. In addition, the Repository sends two copies of the PL to the AR at the time of shipment. The AR confirms with the recipient that the entire shipment was received in good condition, then enters the date of receipt and signs the PL in the space indicated to confirm receipt. The AR must return a copy of the signed PL to SMO within seven days of shipment receipt. The second copy of the PL is retained for the AR's files.

**CONTAINERS SUPPLIED THROUGH THE  
USEPA SAMPLE BOTTLE REPOSITORY**

<u>Container Type</u>	<u>Description</u>	<u>Used for Sample Type</u>
A	80-oz. amber glass bottle w/teflon-lined black phenolic cap	Extractable Organics
B	40-mL glass vial w/teflon-backed silicon septum cap	Volatile Organics (Water)
C	1-L high-density polyethylene bottle w/poly-lined, baked poly cap	Metals, Cyanide & Sulfide
D*	120-mL glass vial w/teflon-lined, white poly cap	Volatile Organics (Soil)
E	16-oz. wide-mouth glass jar w/teflon-lined, black poly cap	Ext. Organics & Metals In Soils & Med/High Water
F	8-oz. wide-mouth glass jar w/teflon-lined, black poly cap	(same as Type E)
G	4-oz. wide-mouth glass jar w/teflon-lined, black poly cap	(same as Type E)
H	1-L amber glass bottle w/teflon- lined, black poly cap	(same as Type A)
J	32-oz. wide-mouth glass jar w/teflon-lined, black poly cap	(same as Type E)
K	4-L amber glass bottle w/teflon- lined, black phenolic cap	(same as Type A)
L	500-mL high-density polyethylene bottle w/poly-lined, baked poly cap	(same as Type C)

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\*The NPO recommends the use of container type B, instead of container type D, for volatile organics (soil). A suitable cap liner for container type D is currently under consideration.

#### **4. Summary of Container Cleaning and Quality Control Procedures**

Containers provided under this program are cleaned in Lots of approximately one hundred containers. (Exact Lot sizes for each container type are determined as a multiple of a case so that a container Lot is not split between cases.) Each container Lot is assigned a unique identifying number. This Lot number is permanently affixed to each container in the Lot, recorded in the Repository logbook, and entered on the PL when containers from that Lot are shipped. For quality assurance purposes, each container's Lot number must be permanently associated with the sample collected in that particular container. Samplers should record each container Lot number and associated CLP sample numbers in their field records at the time of sample collection.

The Repository routinely performs QC analyses on one percent of the number of containers per Lot. If a container fails to pass the QC test(s), the associated Lot of containers is reprocessed through the system. No Lot is released for shipment until acceptable QC results are verified.

An additional container is removed from each Lot and stored for QC purposes. QC storage containers are kept in a contaminant-free area of the Repository which is monitored for volatile compounds. The QC storage containers are retained for one year in order to recheck for cleanliness should possible contamination of a Lot of containers come into question at a later date.

A QC release number, assigned to each Lot of containers that passes QC analysis, is marked on both the analysis and storage QC containers for each Lot. The QC release number is cross-referenced with the Lot number in Repository records, so that all QC records can be accessed based on the Lot number identification.

#### **5. Procedures for Problem Resolution**

##### **a. Resolving Problems Concerning Container Shipment**

If there are problems relating to shipment (i.e., shipment does not arrive by scheduled date, shipment is incomplete, or contents are damaged), the AR or shipment recipient (as appropriate to the situation) should contact the Repository immediately to resolve the problem. If the problem is not satisfactorily handled, the AR should then contact SMO for resolution.

##### **b. Resolving Problems Concerning Container Contamination**

If a user has definitive cause to suspect that container contamination may affect sample analysis results, the concerned RSCC should notify SMO by telephone and follow up with an explanatory memorandum directed to the Repository PO and copied to SMO. The memorandum should include a description of the problem, rationale for suspecting container contamination, supporting documentation (if available), and Lot number(s) for all containers concerned. The user should verify that the contamination encountered is not a result of

either improper field procedures (e.g., use of contaminated water for field blanks) or poor laboratory practice (e.g., background contamination) and include this information as part of the rationale in the memorandum submitted to the PO.

Upon PO request, the Repository will check the QC analysis record for the concerned Lot(s) of containers and verify that contract procedures were correctly followed and that the Lot passed the QC analysis. Should an error be identified in this process, the Repository will immediately notify SMO.

As a second step, following PO authorization, the Repository will pull the QC storage container for the Lot(s) and analyze the container(s) for suspected contaminants. SMO will notify the RSCC of the analysis results so that if there is a contamination problem, analysis data from samples collected in other containers in that Lot can be appropriately flagged. Should contamination be confirmed by analysis of the QC storage container, the Repository will immediately identify the problem and correct procedures as necessary to resolve it. Should a wide-spread problem be identified at any time, SMO would notify ARs in a timely manner so that affected containers could be pulled before use in the field.

## **B. Shipment Management Program**

The Shipment Management Contractor establishes, maintains and monitors all shipping accounts for the transportation of CLP materials. Currently, the Contractor coordinates accounts for the shipment of sample containers, sample coolers and contract compliance screening results. Other items that are routed for CLP use may also be addressed by this program at the request of the NPO.

### **1. Sample Containers**

A packing list accompanies all cases of containers that are shipped from the Sample Bottle Repository to designated recipients. At the time of shipment, the Repository sends a copy of the packing list to the Shipment Management Contractor who utilizes the list to verify and pay shipping invoices. SMO notifies the Contractor of any shipments that require special tracking (e.g., shipments for overnight delivery, shipments not originating at a Repository). If any questions arise regarding the shipment of sample containers, the Contractor contacts the appropriate Repository for resolution.

### **2. Sample Coolers**

Field samplers package samples into coolers for transportation to contract laboratories per the procedures specified in Chapter III, Section D. Sampling contractors are responsible for clearly marking a return address on the outside of each cooler. Contract laboratories are required to return each cooler to the indicated sampling office within fourteen days of sample receipt. The Shipment Management Contractor is responsible for tracking and paying for cooler shipments from the laboratories to the sampling offices.



### **3. Contract Compliance Screening Results**

After reviewing each data package via the Contract Compliance Screening (CCS) process (see Section G), SMO distributes the results to EMSL/LV, the appropriate Region and the relevant laboratory. SMO also sends a copy of the air carrier manifest to the Shipment Management Contractor who uses the manifest to verify and pay shipping invoices. If any problems arise regarding the shipment of CCS results, both SMO and the Shipment Management Contractor should be notified immediately.

### **C. Environmental Services Assistance Teams**

ESAT contracts provide technical, management and other related resource support for Superfund and nonSuperfund Agency programs. The two ESAT contracts are defined by zones with ESAT Zone 1 supporting Regions I, II, III and V, and ESAT Zone 2 supporting EPA Headquarters and Regions IV, VI, VII, VIII, IX and X.

ESAT contractors provide assistance in the following task areas: 1) analytical support including chemical analysis and data reporting per CLP or other designated methods; 2) review of CLP and other analytical data to determine data quality for purposes of usability; 3) logistical and administrative support including sample, data and document control; 4) QA/QC support including preparation and review of QA project plans, CLP special analytical services method definition, and CLP IFB protocol review; 5) management and reporting; and 6) other task related activities to be defined through EPA technical direction as the needs occur. Unless otherwise directed, ESAT contractors apply CLP protocols and follow program guidelines.

### **D. Information Services**

#### **1. Regional Backlog Inventory Report**

Upon request, SMO distributes a Regional Backlog Inventory Report to the DPO. This computerized report provides a summary of the Region's use of CLP resources during a specified time period. The following information is included in the Backlog Inventory Report:

- o Case number
- o Sample number
- o Laboratory name and contract number
- o Laboratory sample receipt date
- o Sample weight and components analyzed
- o Sample type
- o Data due date

- o Days late/early calculations for contractually required deliverables (i.e, extraction, VOA analysis and sample data package)
- o Invoice numbers
- o CCS results to lab date
- o Data complete date

The Region utilizes the Backlog Inventory Report for management and resource planning as well as verifying monthly sample receipts and analyses performed. An example of the Regional Backlog Inventory Report is contained in Appendix E.

## **2. Sample Status Information**

After scheduling analysis, SMO tracks samples from shipment through data reporting via manual and computerized tracking systems. SMO maintains ongoing communication with the DPOs, RSCCs and laboratories regarding sample status and responds to inquiries from concerned parties, as appropriate. A backlog report listing each laboratory's samples and the number of days the samples have been in-house is sent bimonthly to the DPOs and laboratories.

## **3. General Program Information**

Under the direction of CLP management, SMO serves as the program's information center for incoming calls and correspondence. Upon request, SMO provides program participants and interested parties with information and materials on program services and procedures, and refers callers to the proper sources for additional information.

# **E. Enforcement Support**

## **1. Generation of Enforcement Quality Data**

One major objective of Superfund is to recover costs incurred in the investigation and clean up of hazardous waste sites from responsible parties. The process by which these parties are identified and determined to be responsible often involves litigation. Frequently, the Agency's case uses CLP data generated from the analysis of samples collected at a given site. The CLP supports these and other enforcement requirements of Superfund by ensuring that CLP analytical data is documented and available for litigation. Through NEIC, the CLP has established detailed procedures and documentation to ensure that sample data meet Agency enforcement standards.

### **a. Chain-of-Custody and Document Control**

Each CLP analytical contract requires the laboratory contractor to implement a comprehensive document control system and to employ strict chain-of-custody procedures in the receipt and handling of samples throughout the analytical and data reporting process. The laboratory must have written standard operating procedures for receipt and log-in of samples,

maintenance of sample security after log-in, tracking of samples through all steps of preparation and analysis, and organization and assembly of all sample-related documentation on a Case-specific basis. At a minimum, required document control and chain-of-custody records include custody records, sample tracking records, analyst logbook pages, bench sheets, chromatographic charts, computer printouts, raw data summaries, instrument logbook pages, correspondence and document inventory.

Before a laboratory is awarded a CLP contract and continuing periodically throughout the life of the contract, NEIC audits each laboratory facility to ensure compliance with chain-of-custody and document control requirements. In addition to facility audits, NEIC reviews laboratory data and evidence documentation on a regular basis.

b. NEIC Evidence Audits

Laboratories are contractually required to submit a complete Case file purge package, containing all evidence and other documentation relating to sample analysis, within 270 days after submission of analytical data. The Contractor Evidence Audit Team (CEAT) reviews all Case file purge packages to verify that the documentation is complete and conforms to contractual requirements; CEAT routinely audits a selected number of packages to determine adherence to procedure. Following review and/or audit, NEIC sends laboratory Case file purge packages to the Region, where the packages are filed with the analytical data and may be subject to additional review. A list of Case file purge materials is included in Appendix E.

NEIC evidence audits may involve production of sample profiles. A sample profile traces the path and handling of specific samples from the point of collection through shipping, laboratory receipt, chemical analysis and data reporting. The profile identifies all evidence and sequence of events necessary to reconstruct the sample history and thus present to the case attorney a depiction of the sample integrity. In addition to the routine generation of sample profiles in evidence audits, authorized Regional personnel and enforcement attorneys may request NEIC to prepare Case-specific sample profiles to support enforcement activities.

**2. Additional CLP Enforcement Support**

Court appearances and other mandated deadlines often do not allow sufficient time for completion of the normal Case file purge package submission, review and audit process. In this event, enforcement activities require direct CLP support. Data package evaluation and/or testimony from laboratory or CLP personnel may also be needed. Through SMO, the CLP has established procedures to coordinate and respond to short term enforcement-related requirements.

a. Request Procedures

Regional counsel, NEIC or other designated EPA personnel submit enforcement-related requests in a memorandum to the NPO. The NPO reviews the memorandum, determines

necessary CLP action and forwards the request along with directions for action to SMO. If a request requires immediate response, the requestor should contact SMO directly by telephone and follow up with the written request memorandum to the NPO.

**b. Requestor Information Required**

To initiate CLP action, the following information must be provided by the requestor:

- o Name and telephone number of Regional contact coordinating the enforcement activity
- o Case/SAS number(s) of specific site sampling(s)
- o Sample number(s)
- o Date(s) of sample collection
- o Laboratory(ies) that performed the analysis
- o Type of support needed

Most requests can be met quickly; however, a two week lead-time is strongly recommended.

**c. Documentation/Support Provided by CLP**

In responding to enforcement-related requests, SMO provides the following support:

- o Arranges for the timely delivery of all laboratory and evidence documentation relating to specific sample analyses (within a minimum of seven days of request, if designated).
- o Obtains information relating to sample analysis or handling not specifically required under laboratory contracts.
- o Assists in arranging for expert testimony by laboratory or CLP personnel.
- o Augments Regional resources for analytical data review.

## **F. Cost Recovery Substantiation**

The CLP provides documentation for program analytical costs to the EPA's Office of Waste Programs Enforcement (OWPE) in support of Superfund cost recovery efforts.

### **1. Request Procedures**

Requests for cost recovery documentation must be made by completing a Cost Recovery (CR) checklist and mailing it to OWPE. This checklist is designed to provide basic site information needed to compile cost documentation from the CLP and other sources. A copy of the OWPE CR checklist is included in Appendix E.

In response to requests, OWPE collects and organizes cost-related documentation from the CLP and several other sources, such as the EPA Financial Management Division, the EPA

Office of Solid Waste and Emergency Response, and REM, FIT, TAT and other Agency contractors. In case of conflicts, OWPE is responsible for prioritizing incoming requests. A minimum lead-time of four to six weeks is required to provide the requestor with a full site cost recovery report.

## **2. Requestor Information Required**

To enable the CLP to prepare its cost documentation package, requestors must supply the following information on the CR checklist:

- o Identification number. The appropriate CLP Case or SAS number must be entered here. Although rare, if the Case or SAS number refers to more than one site, the specific sample numbers (from the Case Traffic Reports or SAS Packing Lists) related to the sites in question must be provided.
- o Name and location of site.
- o Date the cost report is needed. A minimum of four weeks from the date of request must be given. Six week lead-time is recommended whenever possible.

## **3. Documentation Provided by CLP**

The following information is assembled by SMO and submitted to OWPE:

- o Financial Summary for Cost Analysis—This summary lists analytical and sample management costs on a Case and/or SAS basis and shows total expenses for a particular site. Information on how sample management costs are computed is included.
- o Summary of Invoices, Vouchers and Canceled Checks—This report lists all SAS laboratory invoice numbers and includes SAS canceled check numbers. The summary is organized by SAS number and laboratory name.
- o Routine Analytical Services Cost Report—This computerized report is organized by Case number and laboratory contract. The report includes laboratory invoice numbers, net analysis costs, total of adjustments for contractual noncompliance, early delivery considerations, and sample management costs; and lists total costs on a sample-by-sample, laboratory contract and Case basis.
- o Routine Analytical Services Case Sample List—This computerized report is organized by Case number and laboratory contract with laboratory invoice references. The report provides detail on deliverable turnaround times, analysis components and sample types.
- o Special Analytical Services Cost Report—This computerized report provides a brief description of the service provided and includes the number of samples analyzed, data turnaround time, contract start date, laboratory receipt date, unit costs sample management costs, and contract status. The report also lists total contract costs on SAS and laboratory bases.
- o Copies of all SAS-Related Canceled Checks and Laboratory Invoices.

OWPE provides this CLP information along with documentation gathered from other sources to the Regional case development team in the full cost recovery package.

### **G. Contract Compliance Screening**

SMO performs CCS on all RAS data produced by the CLP. Modified CCS can also be performed on a case-by-case basis on "RAS Plus SAS" or "All SAS" data.

CCS is a structured review which determines completeness of data deliverables and compliance of QA/QC parameters with contract specifications. The primary objectives of CCS are to resolve identified discrepancies in a timely manner and to identify the liquidated category for data not in compliance. Data which meet all CCS criteria at initial receipt are recommended for 100% payment of the amount due. Data with CCS defects have some payment recommendation withheld, either temporarily or permanently, depending on the nature and extent of the defect identified.

Structurally similar CCS procedures are applied to organic, inorganic and dioxin data. CCS results are produced on a fast-turnaround basis (fifteen days) and identify compliance discrepancies by code, criterion, fraction and sample. Results are distributed to the relevant laboratory, Region and EMSL/LV.

Results are accumulated in the CCS Database in order to produce routine and requested summaries of laboratory performance and compliance trends. Examples of CCS result forms are included in Appendix E.

### **H. Data Review Services**

A full range of review services are used to assess CLP data. Objectives of the review services are:

- o To determine the usability and limitations of data given particular field or policy assessment criteria.
- o To maximize the amount of usable data by identifying critical properties of data and by resolving or proposing solutions to analytical or quality control problems.
- o To provide systematic and standardized data quality assessment and status summary to determine method, laboratory and program performance.

These review services are performed by a number of operations:

- o Review for data usability is performed by Regional personnel and contractors. Recommended review procedures have been standardized and organized into functional guidelines for evaluating CLP data. EPA Data Validation Workgroups have produced specific documents for review of organic, inorganic and dioxin analyses.

- o Comprehensive QA review is performed by EMSL/LV on specific data packages. Review and assessment of some program-wide QA results are also performed by EMSL/LV to evaluate method and laboratory performance and the quality of analytical data.
- o Under direction of the CLP management, EMSL/LV and/or SMO may perform additional data review to assess a problem Case or provide a second opinion on usability.

All requests for SMO data review services should be placed using the SMO Data Review Request memorandum available from SMO. An example of this memorandum is provided in Appendix E. Copies of the request should be submitted to SMO (Attention: Data Review Team), the SMO PO and the RSCC. Upon authorization by the PO, SMO schedules the review and notifies the requestor of the date of scheduled completion. (Data review cannot be initiated until all deliverables for the subject Case(s) have been received from the laboratory.)

#### **1. Requestor Information Required**

In completing the Data Review Request form, the client must provide the following information for each Case:

- o SMO Case number
- o Site name
- o Analytical laboratory name(s)
- o Number of samples
- o Sample list
- o Type(s) of review requested
- o Requested date for review completion
- o User name and contact
- o Intended use of data

A minimum lead-time of two weeks is required for data review. However, review time is variable depending upon the number of samples involved and the nature of the review. If conflicts occur, the appropriate DPO(s) will be notified and asked to prioritize requests.

#### **2. Documentation Provided by CLP**

An evaluation report that includes a sample/result matrix and supporting statistics and documentation is produced for each type of review. For each sample fraction, the report indicates whether the data are considered acceptable, acceptable given qualifications noted or unacceptable. Reasons for the designation are discussed and completed data review forms for each of the areas of performance are included in the report to the client.

## **CHAPTER V**

### **LABORATORY SELECTION AND STARTUP**



## A. Laboratory Selection Process

### 1. Qualification Requirements

#### a. Preaward Performance Evaluation Sample Analysis

The first criterion for laboratory selection is preaward performance evaluation (PE) sample analysis. Laboratories request preaward PE samples through the Contracting Officer (CO) and, if required, submit a deposit that is returned upon submission of the PE sample data results.

PE samples, distributed by EMSL/LV, are representative of the types of field samples that the laboratory would be routinely analyzing under the subject procurement. The laboratory is required to analyze PE samples according to contract procedures set forth in the IFB and to report PE sample data according to IFB requirements. The standard turnaround time for PE sample data submission is twenty-one days. Bidders' PE sample data are evaluated by NPO and EMSL/LV personnel for compliance with contract requirements and accuracy of determination of compounds at the levels known to be in the PE samples. Analysis results are rated by a scoresheet developed by the NPO and EMSL/LV; currently, the acceptable performance score is seventy percent.

#### b. Bid Price

The second criterion for laboratory selection is bid price. Following bid opening, bid abstracts are reviewed and evaluated by NPO and Contracts Management Division (CMD) officials. The lowest competitive bidders that have received acceptable performance scores for their PE samples are evaluated for bidder responsibility as detailed below.

### 2. Bidder Responsibility

#### a. Bidder-Supplied Documentation

At the time of submission of PE sample data, bidders are required to submit documented evidence that they have the internal procedures, equipment and personnel in place for successful performance of contract requirements. Required documentation includes: 1) functional descriptions and detailed resumes of key personnel, 2) inventory of laboratory equipment and description of laboratory space, and 3) written Standard Operating Procedures (SOPs). Submitted documentation is reviewed by NPO and EMSL/LV personnel and is utilized by the EPA in performance of the site evaluation. After contract award, bidders are required to submit revised SOPs to the PO.

#### b. Laboratory Site Evaluation

NPO, CMD, EMSL/LV and NEIC personnel participate in onsite evaluations of laboratory facilities of bidders which scored acceptably on the PE sample analyses and are within the EPA-determined competitive range. The results of the onsite evaluation are considered in the final determination of bidder responsibility for contract award.

**B. Laboratory Startup Process**

Laboratories entering the program undergo a learning curve process during which they become fully familiarized and obtain expertise in the application of program methodologies and QC procedures. To reduce the learning curve period, the CLP utilizes a series of laboratory startup procedures during the laboratory's initial contract operations and whenever problems are identified during contract performance.

**1. Provision of Standards to Laboratory**

Immediately following contract award, laboratories are required to order analytical reference standards from the Agency's contractor-operated QA Materials Bank. These standards are used by the laboratory to verify laboratory supplied standards throughout contract performance. Chapter VI, Section A provides further information on analytical standards.

**2. PO Review of First Data Packages**

Initial data packages are targeted for immediate review and evaluation by the PO, EMSL/LV and the Region. This intensive review focuses on any problems the laboratory may have in applying methodologies or in reporting data. The PO and DPO supply feedback to the laboratory concerning the status of the data and work with the laboratory in identifying and remedying problems. Depending on the extent of the problems found during the review of an initial data package, the PO or DPO may visit the laboratory facility and work onsite with laboratory personnel to rectify problems.

**3. PO/DPO/SMO/Laboratory Communication**

Telephone communication is the most widely applied method for problem-solving and maintaining efficient laboratory operations during both the laboratory startup phase and throughout the performance of the contract. In general, the laboratory notifies SMO immediately upon identification of any problem regarding the samples or any difficulties encountered in analysis. SMO routinely resolves sample-related problems in coordination with the Regional client and refers technical problems to the PO or DPO, who then contacts the laboratory to resolve the problem. The resolution and any specific actions taken are reported to the appropriate SMO personnel who records this information as part of the permanent Case record. The laboratory also records the problem and resolution in the narrative portion of the sample data report so that the Region will consider this information when evaluating and using the data.

**C. Laboratory Performance Evaluation****1. Performance Evaluation Sample Analysis**

On a quarterly basis, EMSL/LV distributes PE samples to contract laboratories for analysis. EMSL/LV then evaluates the laboratories' PE sample data, and the NPO uses this evaluation

in formally assessing laboratory contract performance. Additionally, EMSL/LV enters PE sample data into the program's QA and Results Database. These data are utilized, along with other laboratory data, in trend analyses and evaluation of contract QC criteria. Refer to Chapter VI, Section C for a more detailed description of PE samples.

## **2. Onsite Laboratory Evaluation**

Regional, NEIC and EMSL/LV personnel visit each contract laboratory facility in order to evaluate laboratory procedures. The frequency of onsite evaluation depends, in part, upon laboratory performance. The NPO utilizes the evaluation reports which result from these onsite visits in identifying and remedying laboratory performance problems. Chapter VI, Section E details the onsite laboratory evaluation process.

## **3. Corrective Action**

The PO and DPO work closely with each laboratory to correct identified laboratory performance problems. Depending on the scope of the problems, the laboratory may be placed on temporary hold and will not receive additional samples for analysis until the problem has been corrected.

If the laboratory's noncompliance to contract performance or delivery requirements continues, the NPO may request the CO to initiate a contract action such as a Show Cause Notice. A Show Cause Notice requires the Contractor, within a ten-day period, to present any facts the Government can use to determine if the Contractor's failure to perform arose without any fault or negligence on the part of the Contractor. The Contractor must submit substantial evidence to demonstrate that the contract should not be terminated for default.

A recovery plan is generally included as part of the Contractor's response to the Show Cause Notice. NPO and CMD officials review the Contractor's response and proposed recovery plan to determine whether the Contractor has presented sufficient evidence to demonstrate timely remedy of the noncompliance. Following this review, if the Contractor has presented acceptable evidence toward recovery, the Government issues a Cure Notice to the Contractor. A Cure Notice delineates the Government-accepted recovery plan that the Contractor must follow to avoid contract termination. The recovery plan includes actions and time schedules for completion of each step of the recovery process, and specifies an overall time period acceptable for completion of recovery.

Should the Contractor not comply with the recovery schedule, the Government's next and final step may be contract termination for default. In addition to terminating the laboratory's contract, this action affects the evaluation of the laboratory's responsibility for award under future CLP solicitations.

## **CHAPTER VI**

### **PROGRAM QUALITY ASSURANCE**

Quality assurance (QA) and quality control (QC) are integral parts of the CLP. The QA process consists of management review and oversight at the planning, implementation, and completion stages of environmental data collection. The QA process ensures that the data provided is of the quality required. The QC process includes the activities required during data collection to produce the data quality desired and to document the quality of the collected data.

During the planning of an environmental data collection program, QA activities focus on defining data quality criteria and designing a QC system to measure the quality of data being generated. During the implementation of the data collection effort, QA activities ensure that the QC system is functioning effectively, and that the deficiencies uncovered by the QC system are corrected. After environmental data are collected, QA activities focus on assessing the quality of data obtained to determine its suitability to support enforcement or remedial decisions.

A complete QA/QC program includes internal laboratory QC criteria that must be met at acceptable levels of performance. These performance levels are determined by QA review. External review of data and procedures is accomplished by the monitoring activities of the NPO, the Regions, SMO, NEIC and EMSL/LV. Blind performance samples, magnetic tape audits and laboratory onsite evaluations provide an external QA reference for CLP. A feedback loop supplies the results of the various review functions to the contract laboratories through direct communication with the POs and DPOs. The following sections describe overall QA/QC operations and how the CLP meets the QA/QC objective.

### **A. Laboratory Quality Control Criteria**

#### **1. Standard Operating Procedures**

In any operation that is performed on a repetitive basis, assurance of data quality and reproducibility is best accomplished through the use of SOPs. All SOPs, as prepared and presented to the Agency by the Contractor, reflect activities as they are currently performed in the laboratory. In addition, laboratory SOPs:

- o Are consistent with current EPA regulations, guidelines, and CLP contractual requirements.
- o Are consistent with instrument manufacturers' specific instruction manuals.
- o Are available to EPA personnel during an onsite laboratory evaluations.
- o Provide documentation that is sufficiently complete to record the performance of all tasks required by the analytical protocol.
- o Demonstrate the validity of data reported by the Contractor and explain the cause of missing or inconsistent results.
- o Describe the corrective measures and feedback mechanisms utilized when analytical results do not meet protocol requirements.
- o Are updated as necessary when contract, facility, or contractor procedural modifications are made.

- o Are archived for future reference in usability or evidentiary situations.
- o Have the appropriate portions available at the appropriate work stations.
- o Are subject to a document control procedure which precludes the use of outdated or inappropriate SOPs.

The Agency requires SOPs for sample storage and preparation, glassware cleaning, calibration, analytical procedures and standards, maintenance activities, and data reduction, documentation and validation procedures. In addition, evidentiary SOPs are required as stated in each analytical Statement of Work. The SOP format may vary depending upon the kind of activity for which the SOP is prepared.

Following contract award, the laboratory sends a complete set of SOPs to the DPO, EMSL/LV (quality assurance SOPs) and NEIC (evidentiary SOPs). Once SOPs have been submitted, the laboratory is responsible for providing any revised or new SOPs to the DPO, EMSL/LV and NEIC, as appropriate.

## **2. Quality Assurance Plan**

Each contract laboratory establishes a QA program with the objective of providing sound analytical chemical measurements. The program incorporates the QC procedures, any necessary corrective action, and all documentation required during data collection as well as the quality assessment measures performed by management to ensure acceptable data production. As evidence of such a program, the Contractor prepares a written Quality Assurance Plan (QAP) which achieves the following:

- o Maintains data integrity, validity and usability.
- o Ensures that analytical measurement systems are maintained in an acceptable state of stability and reproducibility.
- o Ensures a consistent number of qualified personnel sufficient to meet contract requirements and deliver the product in a timely fashion.
- o Detects problems through data assessment and establishes corrective action procedures which keep the analytical process reliable.
- o Documents all aspects of the measurement process in order to provide data which are technically sound and legally defensible.

The QAP presents the policies, organization, objectives, functional guidelines, and specific QA/QC activities designed to achieve the data quality requirements in the analytical contract. Elements of a QAP include organization and personnel, facilities and equipment, document control, analytical methodology, data generation, and QA/QC. Where applicable, the Contractor includes or references SOPs pertaining to each element as part of the QAP. In addition, the QAP is available during onsite laboratory evaluations. Appendix F contains references relevant to the preparation of a QAP.

### 3. Analytical Standards Traceability Requirements

As an element of overall QA, the Agency has established a repository of analytical standards and calibration materials for use in the CLP. All analytical data generated by the CLP are required to be traceable to EPA Repository standards. Traceability must be applied by the contract laboratories to all calibration and QC solutions used to generate data for CLP requirements. Standards supplied by the EPA Repository are provided for the purpose of traceability only and are not routinely used as working standards. Each contract laboratory is responsible for establishing its own specific standards traceability program.

After contract award, the Contractor is required to request a series of calibration and QC solutions from the EPA Repository. In response, the EPA Repository supplies ampoules containing single or multiple analyte solutions. All ampoules are labeled with a lot number, date of preparation, component concentration(s), and solvent(s). The Contractor retains the EPA Repository standards in such a manner as to preserve their integrity. At present, storage at 4°C is required.

Contract laboratories prepare working standards from material obtained from EPA or from commercial sources. Laboratory working standards are not provided by the EPA Repository. Whenever new laboratory working standards (calibration or QC solutions) are prepared, the Contractor demonstrates equivalence of each batch of standards by providing traceability directly to a dilution of an EPA Repository standard. The EPA Repository standard and the laboratory working standard are analyzed by the conditions specified in the analytical Statement of Work. Verification of traceability includes qualitative and quantitative criteria, and specific requirements are system dependent (i.e. GC, GC/MS).

To demonstrate that the laboratory working standards have not degraded while in use, the Contractor compares the working standard concentration against EPA Repository standards according to the traceability requirements described in the Statement of Work. If the laboratory working standard does not meet the quantitative traceability requirements, a new working standard is prepared.

Records and raw data for all standard solution traceability verification include signed and dated logbooks with sufficient information to trace the analysis of a sample, or analyte, to a specific pair of working and EPA Repository standards. Thus a standard chain-of-custody exists creating documentation that verifies the acceptability of qualitative and quantitative determinations based on a specific standard lot.

#### B. Analytical Data Review

Upon completion of analysis and data reporting, the contract laboratory simultaneously sends a copy of the complete data package to SMO, EMSL/LV and the Regional client. Each of these groups performs complementary aspects of data review. SMO CCS review identifies contractual discrepancies; EMSL/LV review determines technical quality and consistency; and Regional data review relates usability of the data to a specific site.

**1. Contract Compliance Screening**

CCS is one aspect of the Government's contractual right of inspection of analytical data. CCS examines the Contractor's adherence to the contract requirements based on the sample data package delivered to the Agency. CCS results are used in conjunction with other information to measure overall contractor performance and to take appropriate actions to correct deficiencies in performance.

Upon receipt, SMO screens every RAS CLP-generated data package on a fast-turnaround basis. To ensure a uniform review, a set of standardized procedures have been developed to evaluate the sample data package against the technical and completeness requirements of the contract. The following key areas are reviewed for compliance with the contract: holding times, GC/MS tunes, initial and continuing calibrations, blanks, surrogate recoveries, and matrix spikes and matrix spike duplicates.

CCS results are distributed to the Contractor and all other data recipients. If any problems with the data package are identified, the Contractor has a period of time to correct the deficiencies, send all corrections to SMO, EMSL/LV and the Regional client, and include the corrected resubmittals in the purge of their Case files.

**2. EMSL/LV Data Review**

Periodically, EMSL/LV performs a comprehensive QA audit on a subset of CLP sample data packages using a Mil. Standard 105D approach. EMSL/LV also provides data audits and data evaluation, and participates in special projects (e.g., Dioxin Incineration Study, Love Canal Habitability Study) and special requests such as enforcement support, and preparation and evaluation of data review SOPs.

In addition, EMSL/LV and the NPO manage the program's QA and Results Database. This database includes spike recoveries, blanks, duplicates, tuning, calibration, method of standard additions, ICP check, and analytical results. These data are statistically evaluated and utilized to determine and update contract QC acceptance windows for CLP-generated data and to characterize laboratory, method and program performance.

**3. Regional Data Review**

Contract laboratory data are generated to meet the specific needs of the Regional client. In order to verify the usability of data for the intended purpose, each Region reviews data from the perspective of end-user based upon functional aspects of data quality. As the bases for data evaluation, the Region uses general guidelines for data review that have been developed jointly by the Region and the NPO. Individual Regions may augment the basic guideline review process with additional review based on Region-specific or site-specific concerns.



### **C. Quarterly Blind Performance Evaluation Samples**

As a means of measuring contractor and method performance, contract laboratories participate in interlaboratory comparison studies conducted by the Agency. Results from the analysis of PE samples are used by the Agency to verify the contractors' continuing ability to produce acceptable analytical data. The results are also used to assess the precision and accuracy of the analytical methods for specific analytes.

Sample sets may be provided to participating laboratories on a quarterly basis as either single blind (recognizable as PE material and of unknown composition) or double blind (not recognizable as PE material and of unknown composition) samples. Contractors are required to analyze the samples and return the data package and all raw data within the contract required turnaround time.

At a minimum, the results are evaluated for compound identification, quantitation, and sample contamination. Confidence intervals for the quantitation of target compounds are based on reported values using population statistics. Contractors are required to use the NBS Mass Spectral Library to tentatively identify a maximum number of non-target compounds in each fraction that are present above a minimal response. Tentative identification of these compounds, based on contractually described spectral interpretation procedures, is evaluated and integrated into the evaluation process.

If a Contractor performs unacceptably, the PO or DPO will notify the Contractor concerning the remedy for their unacceptable performance. A Contractor may expect, but the Agency is not limited to, the following actions: reduction of the number of samples sent under the contract, suspension of sample shipment to the Contractor, a site visit, a full data audit, and/or analysis of remedial PE samples.

### **D. GC/MS Tape Audits**

In order to accomplish tape audits, the Agency periodically requests the GC/MS magnetic tapes corresponding to a specific Case. Generally, tape submissions and audits are requested for the following reasons: program overview, indication of data quality problems from EMSL/LV, SMO or Regional data reviews, support for onsite audits, and specific Regional requests.

Depending upon the reason for an audit, the tapes from a recent Case, a specific Case, or a performance sample may be requested. Tape audits provide a mechanism to assess adherence to contractual requirements and to ensure the consistency of data reported on the hard copy/floppy diskettes with the data generated on the GC/MS tapes. This function provides external monitoring of program QC requirements and checks contractor adherence to internal QA procedures. In addition, tape audits enable the Agency to evaluate the utility, precision, and accuracy of the analytical methods.

The GC/MS tape includes raw data and quantitation reports for samples, blanks, matrix spikes, matrix spike duplicates, initial calibrations, continuing calibration, BFB and DFTPP associated with the requested Case. In order to reference raw data to the delivered hard copy, the GC/MS tape submission also includes user-generated spectral libraries, extraction laboratory bench sheets, analysts' laboratory notebook pages, and instrumental reference logbook pages associating the tape files to the raw data files.

### **E. Onsite Laboratory Evaluations**

At a frequency dictated by a contract laboratory's performance, the PO or an authorized representative conducts an onsite laboratory evaluation in order to monitor the Contractor's ability to meet contract terms and conditions. The evaluation process incorporates two separate categories, a QA evaluation and an evidentiary audit.

#### **1. Quality Assurance Onsite Evaluation**

QA evaluators inspect contractor facilities to verify the adequacy and maintenance of instrumentation, the continuity of personnel meeting training requirements, and the acceptable performance of analytical and QC procedures. Items that are evaluated include, but are not limited to, the following:

- o Size and appearance of the facility.
- o Quantity, age, availability, scheduled maintenance and performance of instrumentation.
- o Availability, appropriateness, and utilization of SOPs.
- o Staff qualifications, experience, and personnel training programs.
- o Reagent, standards, and sample storage facilities.
- o Standard preparation and traceability logbooks and raw data.
- o Bench sheets and analytical logbook maintenance and review.

Prior to an onsite evaluation, various documentation pertaining to performance of the specific contractor is integrated in a profile package for discussion during the evaluation. Items that may be included are previous onsite reports, PE scores, Regional review of data, Regional QA materials, GC/MS tape audit reports, CCS results, and data trend reports.

#### **2. Evidentiary Audit**

Evidence auditors conduct an onsite laboratory evaluation to determine if laboratory policies and procedures are in place to satisfy evidence handling requirements as stated in the Statement of Work. The evidentiary audit is comprised of the following three activities: the procedural audit, the written SOPs audit, and the analytical project file audit. The procedural audit consists of review and examination of actual standard operating procedures and accompanying documentation. The written SOPs audit determines accuracy and completeness of the written SOPs. The procedural and written SOPs audits are conducted for the following laboratory operations: sample receiving, sample storage, sample identification, sample security, sample

tracking (from receipt to completion of analysis) and analytical project file organization and assembly. The analytical project file evidence audit consists of review and examination of the analytical project file documentation. The auditors review the files to determine the accuracy of the document inventory, the completeness of the file, the adequacy and accuracy of the document numbering system, traceability of sample activity, identification of activity recorded on the documents, and error correction methods.

### **3. Discussion of the Onsite Team's Findings and Corrective Action Reports**

The QA and evidence auditors discuss their findings with the PO/DPO prior to debriefing the Contractor. During the debriefing, the auditors present their findings and recommendations for corrective actions necessary to the contractor personnel.

Following the evaluation, QA and evidentiary audit reports which discuss deficiencies found during the onsite are forwarded to the Contractor. The Contractor must discuss the corrective actions taken to resolve the deficiencies discussed during the onsite visit and in the onsite reports in a letter to the PO, DPO, EMSL/LV (response to the QA report) and NEIC (response to the evidentiary report) within a specified length of time. If SOPs are required to be written or amended, the Contractor must provide the SOPs to the DPO, EMSL/LV (QA/technical SOPs) and NEIC (evidentiary SOPs).

If the Contractor fails to take appropriate corrective action to resolve the deficiencies, a Contractor may expect, but the Agency is not limited to, the following actions: reduction of the number of samples sent under the contract, suspension of sample shipment to the contractor, a follow-up site visit, a full data audit, and/or analysis of remedial PE samples.

### **F. Quality Assurance and Data Trend Analysis**

The QC prescribed in the analytical methods provides information that is continually used by the Agency to assess sample, contractor and program data quality via data trend analysis. Statistical reports that evaluate specific anomalies or disclose trends in many areas are generated from a computerized database. These areas include surrogate spike recovery, matrix spike/duplicate spike recovery, method blanks, GC/MS tuning and mass calibration, initial and continuing calibration data, and other QC and method parameters.

Program-wide statistical results are used to rank laboratories in order to observe the relative performance of each contractor in a given protocol against its peers. The reports are also used to identify trends within laboratories. The results of many of these trend analyses are included in overall evaluation of a contractor's performance, and are reviewed to determine if corrective action or an onsite laboratory evaluation is indicated in order to meet the QA/QC requirements of the contract.

Contractor performance over time is monitored using these trend analysis techniques to detect departures of contractor output from required or desired QC levels, and to provide an early warning of contractor QA/QC problems which may not be apparent from the results of an individual Case.

As a further benefit to the CLP, the database provides the information needed to establish performance-based criteria in updated analytical protocols. The empirical data set produced by contract laboratories is carefully analyzed with the results augmenting theoretical and research-based performance criteria. The result is a continuously monitored set of QC and performance criteria specifications of what is routinely achievable and expected of environmental chemistry laboratories in mass production analysis of environmental samples. These specifications assist the Agency in meeting its objectives of obtaining data of known and documented quality.

## APPENDIX A LIST OF ACRONYMS

AA	Atomic Absorption
AOB	Analytical Operations Branch
AR	Authorized Requestor
B/N/A	Base, Neutral, Acid
CCS	Contract Compliance Screening
CEAT	Contractor Evidence Audit Team
CERCLA	Comprehensive Environmental Response, Compensation and Liability Act
CLP	Contract Laboratory Program
CMD	Contracts Management Division
CO	Contracting Officer
CR	Cost Recovery
CRQL	Contract Required Quantitation Limit
DPO	Deputy Project Officer
DR	Delivery Request
DSR	Dioxin Shipment Record
EMSL	Environmental Monitoring Systems Laboratory
EPA	Environmental Protection Agency
ERT	Environmental Response Team
ESAT	Environmental Services Assistance Teams
FIT	Field Investigation Team
FR	Federal Register
FSCC	Fused Silica Capillary Column
GC/EC	Gas Chromatography/Electron Capture
GC/MS	Gas Chromatography/Mass Spectrometry
HRGC	High Resolution Gas Chromatography
HRMS	High Resolution Mass Spectrometry
HSED	Hazardous Site Evaluation Division
ICP/MS	Inductively Coupled Plasma/Mass Spectrometry
IDL	Instrument Detection Limit
IFB	Invitation for Bid
LCS	Laboratory Control Sample
NBS	National Bureau of Standards
NEIC	National Enforcement Investigations Center
NPM	National Program Manager
NPO	National Program Office
ORD	Office of Research and Development
OSWER	Office of Solid Waste and Emergency Response
OWPE	Office of Waste Programs Enforcement
PCB	Polychlorinated Biphenyl
PE	Performance Evaluation
PEST	Pesticides
PL	Packing List
PO	Project Officer
QA	Quality Assurance
QAP	Quality Assurance Plan
QC	Quality Control
RAS	Routine Analytical Services
REM	Remedial Action Team
RSCC	Regional Sample Control Center
SARA	Superfund Amendments and Reauthorization Act

**List of Acronyms (cont'd.)**

SAS	Special Analytical Services
SDG	Sample Delivery Group
SICP	Selected Ion Current Profile
SIM	Selected Ion Monitoring
SMO	Sample Management Office
SOP	Standard Operating Procedure
SOW	Statement of Work
SV	Semivolatile
TAT	Technical Assistance Team
TCL	Target Compound List
TIC	Tentatively Identified Compound
TR	Traffic Report
VOA	Volatile

## **APPENDIX B**

### **CLP DIRECTORY**

**CLP NATIONAL PROGRAM OFFICE  
DECEMBER 1988**

**USEPA ANALYTICAL OPERATIONS BRANCH (OS-230)**

401 M Street, S.W.  
Room M-2624  
Washington, DC 20460  
202/382-7906 FTS 382-7906

Joan Barnes, Branch Chief  
202/382-7906 FTS/382-7906  
Lynn Beasley, ESAT Project Officer, Regional Operations Section  
202/475-8607 FTS 475-8607  
Emile Boulos, CLP Project Officer, Organics Section  
202/382-7942 FTS 382-7942  
Angelo Carasea, CLP Project Officer, Organics Section  
202/382-7911 FTS 382-7911  
Mike Carter, Chief, Regional Operations Section-SMO Project Officer  
202/382-7909 FTS 382-7909  
Carla Dempsey, QA Coordinator  
202/382-5746 FTS 382-5746  
Joan Fisk, National Organics Program Manager-Chief, Organics Section  
202/382-3115 FTS 382-3115  
Howard Fribush, CLP Project Officer, Organics Section  
202/382-2239 FTS 382-2239  
Michael Hurd, CLP Project Officer, Inorganics Section  
202/382-7908 FTS 382-7908  
Bill Langley, National Inorganics Program Manager-Acting Chief, Inorganics Section  
202/382-7906 FTS 382-7906  
Mary Mahsetky, CLP Program Technician  
Sylvia Miller, Branch Secretary  
Rona Haley, Secretary, Organics Section

**USEPA CONTRACTS MANAGEMENT DIVISION (MD-33)**

Alexander Drive  
Res. Tri. Park, NC 27711

Frank Rzasa, Deputy Director  
919/541-3046 FTS 629-3046  
Marian Bernd, Solicitations and Contract Awards  
202/382-0532 FTS 382-0532  
Janet Simmons, Contract Placement  
919/541-4081 FTS 629-4081  
Larry Presnell, Contract Administration  
919/541-3166 FTS 629-3166



**USEPA ENVIRONMENTAL MONITORING SYSTEMS LABORATORY (EMSL/LV)**

944 East Harmon Avenue

Las Vegas, NV 89109

(Mailing Address: P.O. Box 93478, Las Vegas, NV 89193-3478)

Data To: EMSL/LV Executive Center  
944 East Harmon Ave.  
Las Vegas, NV 89119  
Attn: Data Audit Staff

Llewellyn Williams, Director, QA Division

702/798-2103 FTS 545-2103

Steve Billets, Deputy Director, QA Division

702/798-2609 FTS 545-2609

Jim D. Petty, Chief, QARB, QAD

702/798-2383 FTS 545-2383

Larry Butler, Research Chemist, QARB, QAD

702/798-2114 FTS 798-2114

Edward Kantor, Chemist, QARB, QAD

702/798-2690 FTS 545-2690

Harold Vincent, Chemist, QARB, QAD

702/798-2129 FTS 545-2129

Dave Bottrell, Chemist, QAD (Organics Method Contract)

702/798-2142 FTS 545-2142

William Newberry, Chemist, QARB, QAD (Inorganics Method Contract)

702/798-2167 FTS 545-2167

Gareth Pearson, Director, Exposure Assessment Research Division

702/798-2203 FTS 545-2203

Bob Schonbrod, Exposure Assessment Research Division

702/798-2229 FTS 545-2229

Lou Blume, Exposure Assessment Research Division

702/798-2213 FTS 545-2213

Gene Meier, Director, Adv. Monitoring Division

702/798-2203 FTS 545-2203

**USEPA NATIONAL ENFORCEMENT INVESTIGATIONS CENTER (NEIC)**

Denver Fed. Cntr. 53, E-2

P.O. Box 25227

Denver, CO 80225

303/236-5111 FTS 776-5111

Tom Gallagher, Director

303/236-5100 FTS 776-5100

Carroll G. Wills, Deputy Director

303/236-5120 FTS 776-5120

Ted Meiggs, Assistant Director, Lab Services

303/236-5132 FTS 776-5132

Joe Lowry, Chief, Environmental Chemistry Branch

303/236-9963 FTS 776-5122

Dean Hill, Pesticides

303/236-8138 FTS 776-8138

Gerri Hilden, Evidence Audit (Chain of Custody)  
303/236-5122 FTS 776-5122  
Donald Roche, Audit Rep. (Primary Contact)  
303/236-5122 FTS 776-5122  
Robert Laidlaw, Enforcement  
303/236-5111 FTS 776-5111

**USEPA ENVIRONMENTAL MONITORING SYSTEMS LABORATORY**  
**(EMSL/CINCINNATI)**

26 W. Martin Luther King Dr.  
Cincinnati, OH 45268

Thomas A. Clark, Acting Lab Director  
513/569-7301 FTS 684-7301  
Gerald D. McKee, Acting Deputy Lab Director  
513/569-7303 FTS 684-7303  
Bill Budde, Chief, Adv'd. Instrumentation-CLP Specialist  
513/569-7309 FTS 684-7309  
Ed Berg, Chief, Project Mgmt. Section-Performance Evaluation  
513/569-7325 FTS 684-7325  
James J. Lichtenberg, Chief, Phys. & Chem. Methods Branch  
513/569-7306 FTS 684-7306  
John A. Winter, Chief, QA Branch  
513/569-7325 FTS 684-7325  
Tom Bellar, Research Chemist  
513/569-7512 FTS 684-7512  
Ted Martin, Inorganic Chemist-Methods Development  
513/569-7312 FTS 684-7312

**FIELD CONTRACTORS - MAIN OFFICE:**

**NUS Corporation** (FIT II - East)  
1300 North 17th Street  
Suite 1320  
Arlington, VA 22209  
703/522-8802

Paul Clay  
Tom Centi

**Ecology & Environment** (FIT II-West)  
1700 North Moore Street  
Rosslyn Center  
Arlington, VA 22209  
703/522-6065

Lou Welzel  
Wendell Fields

CDM Federal Programs Corp. (REM II)  
13135 Lee Jackson Mem Hwy  
Suite 200  
Fairfax, VA 22033  
703/968-0900

Gary Dunbar  
Steve Paquette  
Andy Szilagyi

Ebasco Services, Inc. (REM III)  
Zone Program Management Office  
2000 Fifteenth St., North  
Arlington, VA 22201  
703/558-7555

Michael Yates

CH2M Hill, Inc. (REM IV)  
625 Herndon Parkway  
Herndon, VA 22070  
703/471-1441

Kent Robinson

# SAMPLE MANAGEMENT OFFICE DIRECTORY

November 1988

## Mailing Address:

CLP Sample Management Office  
P.O. Box 818  
Alexandria, Virginia 22313  
703/557-2490 FTS 557-2490

## Street Address:

Viar and Company, Inc.  
209 Madison Street, #200  
Alexandria, Virginia 22314  
703/684-5678

**DAVID H. STEWART**  
**SMO PROJECT DIRECTOR**

**Don Trees, Program Manager**

*Data Processing and Scientific Services*

**Peter Isaacson, Project Manager**  
*Scientific Support Groups (SSG)*

**Sa'ad Masri, Project Leader** ..... *Contract Compliance Screening (CCS)*  
*Inorganics & Organics*  
**Dipti Singh, QA Chemist** ..... *CCS Organics*  
**Fida Abdelwahab, QA Chemist** ..... *CCS Inorganics*  
**Paul Ssenyonga, Data Systems Coordinator** ..... *CCS Diskette Deliverables*  
**Harry McCarty, Project Leader** ..... *Data Review & Method Development*  
**Marianne Lynch, Chemist** ..... *Method Development*  
**Bill Eckel, Chemist** ..... *Data Review*  
**Ruth Bleyler, QA Chemist** ..... *Regional QA/QC Support*

**R. Richard Thacker, Program Manager**

*SMO Operations/Management Planning*

**Linda Boynton, Project Manager**  
*Analytical Services Group (ASG)*

**Lulu Eager, Senior Bookkeeper** ..... *SAS Invoice Processing*  
**Maka Grogard, Senior Environmental Program Analyst** ..... *Operations - ASG*  
*Coordination & Implementation of CLP*  
*Sample Scheduling & Tracking Systems*  
**Sean Kolb, Environmental Program Analyst**  
**Lynn Riddick, Environmental Program Coordinator** ..... *Region VIII*  
**Diane Cutler, Environmental Program Coordinator** ..... *Region II*  
**Jeb Livingood, Environmental Program Coordinator** ..... *Region VI, V SAS*  
**Cindy Schreyer, Environmental Program Coordinator** ..... *Region I, V RAS*  
**Susan Barrell, Environmental Program Coordinator** ..... *(TBA)*

*(Analytical Services Group continued)*

**Carol Shaeffer, Environmental Program Analyst**

Terri Shaughnessy, Environmental Program Coordinator.....*Region III*

Karen Elm, Environmental Program Coordinator..... *Region IX*

Tom Sigler, Environmental Program Coordinator..... *Region IV, VII*

Anne Babyak, Environmental Program Coordinator.....*(TBA)*

**Leslie Braun, Environmental Program Analyst**..... *Management Information Systems - ASG*

Hoang Ho, MIS Coordinator ..... *SAM & SAS Data Base*

Sally Boyar, MIS Coordinator ..... *Contract Funding Status System &  
Distribution of Program Lists*

Michelle Kosloski, MIS Coordinator .....*Extract Disposal System &  
Backlog Inventory*

**Deborah Miller, Project Manager**  
*Program/Contracts Support Group (P/CSG)*

**Mike Tindle, Environmental Program Analyst**.....*Computer Systems Documentation,  
IFB/Procurement Support*

Talia Peters, Sr. Environmental Program Coordinator..... *IFB/Procurement Development*

Heidi Janss, Environmental Program Coordinator .....*Computer Systems Documentation  
IFB/Procurement Support*

Susan Wilkins, Environmental Program Coordinator .....*Meeting Coordination  
IFB/Procurement Support*

Peter Ziu, Environmental Program Coordinator ..... *Bottle Repository Support,  
Automated Address System*

Cheryl Shriver, Environmental Program Coordinator..... *Meeting Coordination  
Materials Distribution*

**Tina DeYoung, Project Manager**  
*Management Information Group (MIG)*  
*(Ad Hoc Requests & Annual Site/Cost Accounting)*

Tina Rodgers.....*DPO Reports & Blue Book Reports*

**Pam Werntz Simons, Management Information Systems Manager** ..... *Site/Cost Accounting,  
Invoicing, Reconciliation, Closeout, Data Control*

Rhonda Harmon..... *Task Manager, Payment Activities*

John Reynolds.....*Team Leader, Payment Processing*

Marta Meixner .....*Team Leader, Payment Processing*

## **USEPA REGION I**

**USEPA Region I, ESD  
60 Westview Street  
Lexington, MA 02173  
617/860-4320**

**Mary Ann Becker, Communications Contact (ESAT)  
617/860-4612  
John Carlson, ESAT DPO  
617/860-4320  
Edward Conley, Director, ESD  
617/860-4320  
Elio Goffi, Communications Contact (Alt)  
617/860-4630  
Vicki Howell, Communications Contact (Alt - ESAT)  
617/229-2050  
Thomas Spittler, Chief, Technical Support Branch  
617/860-4320  
Deb Szaro, Technical DPO, Communications Contact  
617/860-4312**

**USEPA Region I, WMD  
J.F. Kennedy Federal Bldg  
Boston, MA 02203  
(Data Submission: JFK Federal Bldg; Room 1903)**

**Rosalie Baldassari, Primary RSCC-Data Submission  
617/573-5798 FTS 833-1798  
Merrill Hohman, SF Coordinator-Director, Waste Mgmt. Div.  
617/573-5700 FTS 833-1700  
Susan Willis, Non-Primary RSCC  
617/573-5607 FTS 833-1607**

**Camp Dresser & McKee, Inc. (REM II)  
1 Center Plaza  
Boston, MA 02108  
617/742-5151**

**Jim Occhialini**

**Ebasco (REM III)  
211 Congress St.  
8th floor  
Boston, MA 02110-2410  
617/451-1201**

**Russ Boyd  
Lee Dixon**

**USEPA Region I (cont'd)**

NUS Corporation (FIT II)  
19 Crosby Drive  
Bedford, MA 01730  
617/275-2970

Martha Lee

## **USEPA REGION II**

**USEPA Region II  
26 Federal Plaza  
New York, NY 10278**

**Stephen Luftig, SF Coordinator-Director, Emerg. & Remed. Response  
212/264-1574 FTS 264-1574**

**USEPA Region II, ESD  
Woodbridge Avenue  
Building 209  
Edison, NJ 08837**

**Darvene Adams, Non-Primary RSCC  
201/321-6705 FTS 340-6705  
Lou Bevilacqua, Chief, Toxic & Haz. Waste-Technical DPO  
201/321-6702 FTS 340-6702  
Lisa Gatton-Vidulich, Technical DPO (Alt)  
201/321-6676 FTS 340-6676  
Stelios Gerazounis, Dioxin Contact  
201/321-6778 FTS 340-6718  
Amelia Jackson, Organics Contact  
201/321-6164 FTS 340-6164  
Leon Lazarus, Data Review-Organics Contact (Alt)  
201/321-6778 FTS 340-6778  
Dan Lillian, Chief, Tech. Support Branch-ESAT DPO-Lab Director  
201/321-6707 FTS 340-6707  
Gayatri Mehta, ESAT Non-Primary RSCC  
201/321-6705 FTS 340-6705  
Frank Messina, Inorganics Contact  
201/906-6170 FTS 340-6170  
Barbara Metzger, Director, ESD  
201/321-6754 FTS 340-6754  
Regina Odubo-Sullivan, ESAT Primary RSCC  
201/321-6705 FTS 340-6705  
Laura Scolise, Inorganics Contact (Alt)  
201/906-6717 FTS 340-6717  
Richard Spear, Chief, Surv. & Monitoring Branch-Data Submission  
201/321-6685 FTS 340-6685  
Sharon Steltz, Primary RSCC  
201/321-6705 FTS 340-6705  
Dan Sullivan, Deputy Director, ESD  
201/321-6755 FTS 340-6755**



**USEPA Region II (cont'd)**

**NUS Corporation (FIT II)  
Raritan Plaza III  
Fieldcrest Avenue  
Edison, NJ 08837  
201/225-6160**

**Roberta Riccio, Primary Sampling Contact**

## **USEPA REGION III**

**USEPA Region III, CRL  
839 Bestgate Road  
Annapolis, MD 21401  
301/266-9180**

**John Austin, Organic Chemist  
301/266-9180  
Diana Baldi, ESAT DPO-Dioxin & Organics (Alt) Contact  
301/266-9180  
Dan Donnelly, Acting Chief, Lab Section  
301/266-9180  
Jeanne Hankins, Inorganics Contact  
301/266-9180  
Patricia Krantz, Section Chief, QA/QC-Data Submission  
301/266-9180  
Annette Lage, Non-Primary RSCC  
301/266-9180  
Chuck Sands, Technical DPO-Organics & Dioxin (Alt) Contact  
301/266-9180  
John Scalera, Organics Contact (Alt)  
301/266-9180  
Orteria Villa, Jr., Director, CRL  
301/266-9180  
Colleen Walling, Primary RSCC  
301/266-9180  
Claudia Walters, Inorganics Contact (Alt)  
301/266-9180**

**USEPA Region III, SF Branch  
841 Chestnut Street  
Philadelphia, PA 19107**

**Catherine Hodgkiss, Chief, CERCLA Enforcement Section  
215/597-8177 FTS 597-8177  
Green Jones, Director, ESD  
215/597-4532 FTS 597-4532  
Neil Swanson, Acting Chief, CERCLA Remedial Enforcement  
215/597-3186 FTS 597-3186  
Thomas Voltaggio, SF Coordinator  
215/597-8132 FTS 597-8132  
Stephen Wassersug, Director, Waste Mgmt. Div.  
215/597-8131 FTS 597-8131**

**Ebasco Services, Inc. (REM III)  
One Oxford Valley  
Suite 414  
Langhorne, PA 19047-1829  
215/752-0212**

**Carol Chatelain  
Anthony Enweze**

**USEPA Region III (cont'd)**

**NUS Corporation (FIT II)**  
999 West Valley Road  
Wayne, PA 19087  
215/687-9510

Eric Blischke, Primary Requestor  
Russ Sloboda, Chemist-Data Reviewer  
Donna Wallace, Director

**NUS Corporation (REM III - ARCS)**  
Park West Two  
Cliff Mine Road  
Pittsburgh, PA 15275  
412/788-1080

Greg Zimmerman

**Roy F. Weston, Inc. (REM II)**  
1 Weston Way  
West Chester, PA 19380  
213/692-3030

Ralph Shapot

**Weston-SPER (TAT)**  
53 Haddon Field Road  
Suite 306  
Cherry Hill, NJ 08002  
609/482-0222

Bhupi Khona

## **USEPA REGION IV**

**USEPA Region IV  
Superfund Branch  
345 Courtland St., N.E.  
Atlanta, GA 30365**

**Jack Stonebraker, SF Branch Chief  
404/347-2967 FTS 257-2967  
Patrick Tobin, Director, Waste Mgmt. Division  
404/347-3454 FTS 257-3454**

**USEPA Region IV, ESD (ASB)  
Analytical Support Branch  
College Station Road  
Athens, GA 30613**

**Gary Bennett, Secondary Communications Contact  
404/546-3286 FTS 250-3286  
Tom B. Bennett, Jr., Technical DPO-Chief, Org. Chem. Sectn.-Data Submission  
404/546-3112 FTS 250-3112  
Bobby Carroll, ESAT DPO-Chief, ASB  
404/546-3309 FTS 250-3309  
Debbie Colquitt, Non-Primary RSCC  
404/546-3388 FTS 250-3388  
James Finger, Director, ESD  
404/546-3136 FTS 250-3136  
Sandy Fitzgerald, Receptionist  
404/546-3111 FTS 250-3111  
Charles Hooper, Primary Communications Contact  
404/546-3286 FTS 250-3286  
Myron Stephenson, Non-Primary RSCC  
404/546-3385 FTS 250-3385**

**USEPA Region IV, ESD (ECB)  
Env. Compliance Branch  
College Station Road  
Athens, GA 30613**

**Steve Hall, RCRA Team Leader  
404/546-3173 FTS 250-3173  
Doug Lair, Primary RSCC  
404/546-3300 FTS 250-3300  
Doug Mundrick, SF Team Leader  
404/546-3321 FTS 250-3321**

**Camp Dresser & McKee, Inc. (REM II)  
2100 River Edge Parkway  
Suite 400  
Atlanta, GA 30328  
404/952-8643**

**Tom Duffy**

**USEPA Region IV (cont'd)**

**Ebasco  
145 Technology Park  
Norcross, GA 30092-2979  
404/662-2439**

**Loring Pitts**

**NUS Corporation (FIT)  
1927 Lakeside Parkway  
Suite 614  
Tucker, GA 30084  
404/938-7710**

**Phil Blackwell**

## **USEPA REGION V**

### **USEPA Region V, ESD**

**536 S. Clark Street**

**Tenth Floor, CRL**

**Chicago, IL 60605**

**Al Alwan**

**312/353-6619 FTS 353-6619**

**Pat Churilla, Technical DPO-ESAT DPO**

**312/353-9087 FTS 353-908 /**

**Carsten Falkenburg, Communications Contact (Alt - ESAT)**

**312/353-2893 FTS 353-2893**

**Ed Johnson, Organics Contact**

**312/886-5482 FTS 886-5482**

**Duane Kruse, Primary Communications Contact (ESAT)**

**312/353-2893 FTS 353-2893**

**David Payne, Inorganics Contact (Alt)**

**312/886-1973 FTS 886-1973**

**Jan Pels, Primary RSCC**

**312/353-2720 FTS 353-2720**

**Ray Piccione**

**312/886-1974 FTS 886-1974**

**Curtis Ross, Director, CRL-Data Submission**

**312/353-8370 FTS 353-8370**

**William Sanders, III, Director, ESD**

**312/353-3808 FTS 353-3808**

**Nidia Seliciano**

**312/886-0651 FTS 886-0651**

**Jay Thakkar, Inorganics Contact**

**312/886-1972 FTS 886-1972**

**Ira Wilson, Primary Communications Contact (ESAT)**

**312/353-2893 FTS 353-2893**

**Thomas Yeates, Deputy Director, ESD**

**312/353-3808 FTS 353-3808**

### **USEPA Region V, WMD**

**230 S. Dearborn St.**

**13th Floor (HR-13)**

**Chicago, IL 60604**

**312/353-8370 FTS 353-8370**

**Basil Constantelos, Director, Waste Mgmt. Div.**

**312/886-7579 FTS 886-7579**

**William Miner, SF Enforcement**

**312/886-4658 FTS 886-4658**

**USEPA Region V (cont'd)**

**CH2M Hill, Inc. (REM IV)**  
310 W. Wisconsin Ave.  
Suite 700  
Milwaukee, WI 53201  
(Mailing Address: P.O. Box 2090)  
414/272-2426

Jeff Keiser, Non-Primary RSCC (RAS only)  
Dave Shekoski, Non-Primary RSCC (RAS only)  
Shirley Stringer, Non-Primary RSCC (RAS only)

**Camp Dresser & McKee, Inc. (REM II)**  
200 West Adams  
Suite 1600  
Chicago, IL 60606  
312/786-1313

Cynthia Clark, Non-Primary RSCC (RAS only)  
Wendy Dewar, Sampling & Analytical Coordinator  
Jill Line, Non-Primary RSCC (RAS only)

**Ecology & Environment (FIT II)**  
111 W. Jackson Blvd.  
Chicago, IL 60604  
312/663-9415

Tom Clyne, Organics & Dioxin Contact (Alt)  
Zena Gold-Kaufman, Non-Primary RSCC  
Renee Hix-Mays, Sample Coordinator

**Michigan Dept. of Nat. Resources**  
P.O. Box 30028  
Lansing, MI 48909  
517/373-4825

Denise Grubin, Non-Primary RSCC

**Minn. Pollution Control Agency**  
520 Lafayette Road  
St. Paul, MN 55155  
612/296-7735

Dave Kouloski, Non-Primary RSCC  
Becky Lofgrim, Non-Primary RSCC

**USEPA Region V (cont'd)**

**Roy F. Weston, Inc. (TAT)**  
**111 N. Canal Street**  
**Suite 855**  
**Chicago, IL 60606**  
**312/993-1067**

**Eileen Helmer, Non-Primary RSCC (RAS only)**  
**Maureen O'Mara, Non-Primary RSCC (RAS only)**  
**Melodie Sullivan, Non-Primary RSCC (RAS only)**

**Wisconsin Dept. of Nat. Resources**  
**101 South Webster**  
**3rd Floor, GEF-2**  
**Madison, WI 53703**  
**608/267-5063**

**Dick Alberg, Non-Primary RSCC-Organics Contact**  
**Maureen McCurdy, Non-Primary RSCC**  
**Kim McCutchen, Non-Primary RSCC-Inorganics Contact**



## **USEPA REGION VI**

**USEPA Region VI Laboratory  
Monterey Park Pl. Bldg. C  
6608 Hornwood Drive  
Houston, TX 77074  
713/953-3425 FTS 526-9425**

**Diana Ayres, Acting Branch Chief  
713/953-3425 FTS 526-9425  
William Blanton, Communications Contact (ESAT)  
713/953-3425 FTS 526-9425  
Victor Chapman, Communications Contact (ESAT)  
713/953-3425 FTS 526-9425  
Michael Daggett, ESAT DPO-Lab Director-Organics & Dioxin Contact (Alt)  
713/953-3425 FTS 526-9425  
Mahmond El Feky, Inorganics Contact  
713/953-3425 FTS 526-9425  
Harry Kreigh, Communications Contact (Alt - ESAT)  
713/953-3425 FTS 526-9425  
Myra Perez, Primary RSCC  
713/953-3425 FTS 526-9425  
Melvin Ritter, Organics & Dioxin Contact-Data Submission  
713/953-3425 FTS 526-9425  
David Stockton, Technical DPO  
713/953-3425 FTS 526-9425**

**USEPA Region VI, ESD  
Allied Bank Tower  
1445 Ross Avenue  
Dallas, TX 75202  
214/665-6491 FTS 255-6491**

**Allyn Davis, Director, Waste Mgmt. Div.  
214/665-6491 FTS 255-6491  
Charles Gazda, Emergency Response Branch  
214/665-6491 FTS 255-6491  
Dick McGlothlin, Forms  
214/665-6491 FTS 255-6491  
Martha McKee, SF Coordinator  
214/665-6491 FTS 255-6491  
Russell Rhoades, Director, ESD  
214/665-6491 FTS 255-6491  
Hank Thompson, Non-Primary RSCC  
214/665-6491 FTS 255-6491**

**CH2M Hill, Inc. (REM IV)  
6060 South Willow Drive  
Greenwood Vill., CO 80111-5112  
(Mailing Address: P.O. Box 22508; Denver, CO 80222)  
303/771-0900**

**Jane Grogan, CLP Coordinator**

**USEPA Region VI (cont'd)**

**Ecology & Environment (FIT II)**  
**1509 Main Street**  
**Suite 814**  
**Dallas, TX 75201**  
**214/742-6601**

**David Anderson, Non-Primary RSCC (RAS only)**  
**Lloyd Collins, Non-Primary RSCC (RAS only)**  
**Jairo Guevera, Non-Primary RSCC (RAS only)**  
**K. Malone, Regional Program Manager**  
**Gene McDonald, FIT Training Coordinator**  
**John Totin, Asst. Regional Program Manager**

## **USEPA REGION VII**

**USEPA Region VII  
726 Minnesota Avenue  
Kansas City, KS 66101**

**David Wagoner, SF Coordinator-Director, Air & Waste Mgmt. Div.  
913/236-2850 FTS 757-2850**

**USEPA Region VII, ESD  
25 Funston Road  
Kansas City, KS 66115  
913/236-3881 FTS 757-3881**

**Bill Bunn, CLP QA Chief-Comm Contact (Alt)-ESAT DPO  
913/236-3881 FTS 757-3881  
Joyce W. Casper, Primary RSCC-Data Submission  
913/236-3881 FTS 757-3881  
Peggy Cox, Communications Contact (TAT)  
913/236-3881 FTS 757-3881  
Paul Dougherty, FIT RPO  
913/236-3888 FTS 757-3888  
Ron McCutcheon, TAT RPO  
913/236-3881 FTS 757-3881  
Debra Morey, Technical DPO-Communications Contact  
913/236-3881 FTS 757-3881  
Ron Ross, Inorganics Contact (Alt)  
913/236-3881 FTS 757-3881  
Loren Thompson, Communications Contact (ESAT)  
913/236-3881 FTS 757-3881**

**CH2M Hill, Inc. (REM IV)  
6060 South Willow Drive  
Greenwood Vill., CO 80111-5112  
(Mailing Address: P.O. Box 22508; Denver, CO 80222)  
303/771-0900**

**Beth Baruth, CLP Coordinator (Alt)  
Jane Grogan, CLP Coordinator**

## **USEPA REGION VIII**

**USEPA Region VIII Laboratory  
Denver Federal Center  
Box 25366  
Lakewood, CO 80225**

**Alan Curtis  
303/236-5091 FTS 776-5091  
Eva Hoffman, Technical DPO-ESAT DPO-Data Submission  
303/236-7371 FTS 776-7371  
James Lehr, Director, ESD  
303/236-5061 FTS 776-5061  
Deanna Peterson, Primary RSCC  
303/236-7370 FTS 776-7370  
Jon Yeagley, Chief, Lab Services Section  
303/236-5073 FTS 776-5073**

**USEPA Region VIII, ESD  
Denver Place  
Suite 500  
Denver, CO 80202-2405**

**Robert Duprey, Director, Hazardous Waste Management Division  
303/293-1720 FTS 564-1720  
J. William Geise, Jr., Chief, SF Branch  
303/293-1518 FTS 564-1518  
Jay Silvernale, SF Program Section  
303/293-1518 FTS 564-1518  
Judy Wong, SF Enforcement Section  
303/293-1520 FTS 564-1520**

**C.C. Johnson & Malhotra (REM II)  
2300 Fifteenth Street  
Suite 330  
Denver, CO 80202  
303/433-6966**

**Jeff Benson, Dioxin Contact (Alt)  
Bill Berning, Non-Primary RSCC (RAS only)  
Richard Cheatham, Policy Contact-Inorganics & Organics (Alt) Contact  
Jerilyn Guthrie, Non-Primary RSCC (RAS only)**

**USEPA Region VIII (cont'd)**

CH2M Hill, Inc. (REM IV)  
6060 South Willow Drive  
Greenwood Vill., CO 80111-5112  
(Mailing Address: P.O. Box 22508; Denver, CO 80222  
303/771-0900

Dewey Brigham, Non-Primary RSCC (RAS only)  
Jane Grogan, Non-Primary RSCC (RAS only)  
Dennis Neuman, Inorganics Contact (Alt)  
406/994-4822  
Jim Schwing, Regional Manager

Ecology & Environment (FIT II)  
1776 S. Jackson St.  
Suite 200  
Denver, CO 80210-3802  
303/757-4984

Kent Alexander, Dioxin Contact  
Lynn Fischer, Non-Primary RSCC (RAS only)  
Steve Ignelzi, Non-Primary RSCC (RAS only)  
Randy Perliss, Organics Contact  
Stuart Richardson, Regional Program Manager

ICF Technology  
P.O. Box 280041  
Lakewood, CO 80228-2213  
303/236-7412

Regina Rehm, Communications Contact (ESAT)

Jacobs Engineering (TES)  
12600 West Colfax Avenue  
Suite A300  
Lakewood, CO 80215  
303/232-7093

Pam McDevitt, Non-Primary RSCC (RAS only)  
Joyce Miyagishima, Non-Primary RSCC (RAS only)

Montana EPA Office  
301 South Park  
P.O. Drawer 10096  
Helena, MT 59626  
406/449-5414 FTS 585-5414

Mike Bishop, Regional Project Manager  
Eric Fink, Regional Project Manager  
James Knoy, Regional Project Manager  
Liane Shanklin, Remedial Project Manager  
John Wardell, Director, Montana Operations Office

## **USEPA REGION IX**

**USEPA Region IX Laboratory  
944 East Harmon Avenue  
Las Vegas, NV 89119**

**James Johnson, Organics Contact  
702/798-2118 FTS 545-2118  
Ralph Smiecinski, Inorganics Contact  
702/798-2117 FTS 545-2117**

**USEPA Region IX, OPM  
215 Fremont Street  
San Francisco, CA 94105  
(Data Submission: Environ. Serv. Branch, P-3-2)**

**Karen Bankert, Analytical Specialist  
415/974-8856 FTS 454-8856  
David Bingham, Team Leader  
415/974-8149 FTS 454-8149  
Thomas Huetteman, Primary RSCC  
415/974-0923 FTS 454-0923  
Lester Kaufman, Lab Section Chief  
415/974-7484 FTS 454-7484  
Kent Kitchingman, Technical DPO-Data Submission  
415/974-0924 FTS 454-0924  
David Mowday, Deputy Director, OPM  
415/974-8189 FTS 454-8189  
Lilya Rikshpun, Organics & Inorganics Contact (Alt)  
415/974-8801 FTS 454-8801  
Terry Stumph, Chief, ESB-ESAT DPO  
415/974-7483 FTS 454-7483  
Denise Toll, Non-Primary RSCC  
415/974-8004 FTS 454-8004  
Jeff Zelikson, SF Coordinator-Director, Tox. & Waste Mgmt. Div.  
415/974-7460 FTS 454-7460**

**CH2M Hill, Inc. (REM IV)  
2510 Redhill Avenue  
Suite A  
Santa Ana, CA 92705  
714/250-5500**

**Michael Bitner, Project Engineer  
Edward J. Rogan, Project Manager**

**USEPA Region IX (cont'd)**

Camp Dresser & McKee, Inc. (REM II)  
100 Spear Street  
Suite 700  
San Francisco, CA 94105  
415/495-5009

Eric Hinzd, Regional Manager  
John Wondolleck, Principal Engineer

Ebasco Services (REM III)  
One Market Plaza  
Spear Street Tower #600  
San Francisco, CA 94105  
415/777-3000

Dale Rowlison, Regional Manager  
Jim Wilder, Senior Engineer

Ecology & Environment (FIT II)  
160 Spear Street  
14th Floor  
San Francisco, CA 94105  
415/777-2811

P. K. Chattopadhyay, Data Review Coordinator-Dioxin Contact (Alt)  
Ron Karpowitz, FIT Leader  
John Moe, Sampling Advisor

ICF Technology  
160 Spear Street  
Suite 1380  
San Francisco, CA 94105-1535  
415/957-0110

Santiago Lee, Dioxin & Organics (Alt) Contact  
Greg Nicholl, Inorganics Contact (Alt)

## **USEPA REGION X**

### **USEPA Region X Laboratory**

**P.O. Box 549**

**Manchester, WA 98353**

**(Shipping: 7411 Beach Dr. East, Port Orchard, WA 98366)**

**206/442-0370 FTS 399-0370**

**Raleigh Farlow, Secondary Communications Contact**

**206/442-1193 FTS 399-1193**

**Mike Johnston, Chief, Lab Section**

**206/442-0370 FTS 399-0370**

**Gerald Muth, Technical DPO-ESAT DPO-Organics Contact (Alt)**

**206/442-0370 FTS 399-0370**

### **USEPA Region X, ESD**

**1200 Sixth Avenue**

**M/S 329**

**Seattle, WA 98101**

**(Data Submission: Mail Stop ES/096)**

**206/442-1200 FTS 399-1200**

**Robert Courson, Director, ESD**

**206/442-0404 FTS 399-0404**

**Joyce Crosson, QA Specialist-Data Submission-Non Primary-RSCC**

**206/442-2111 FTS 399-2111**

**Charles Findley, Director, Waste Mgmt. Div.**

**206/442-1200 FTS 399-1200**

**Phil Millam, SF Coordinator**

**206/442-1090 FTS 399-1090**

**John Osborn, FIT RPO**

**206/442-0837 FTS 399-0837**

**Kelsey Ramey, Non-Primary RSCC**

**206/442-4323 FTS 399-4323**

**William Schmidt, Chief, Field Oper. & Tech. Support Branch**

**206/442-1526 FTS 399-1526**

**Rhonda Wregglesworth, Primary RSCC**

**206/442-7121 FTS 399-7121**

### **CH2M Hill, Inc. (REM IV)**

**777 - 108th Ave., N.E.**

**Bellevue, WA 98009-2050**

**206/453-5000**

**Stuart Brown**

### **Ecology & Environment (FIT II)**

**101 Yesler Way**

**Suite 600**

**Seattle, WA 98104**

**206/624-9537**

**Andrew Hafferty, Sr Chemist-QA, FIT-Communications Contact**



## MISCELLANEOUS INFORMATION

### Bottle Repositories:

I-Chem Research Corporation (East)  
104 Quigley Blvd.  
New Castle, DE 19720  
302/322-5019

William Luzzo, Repository Manager

Eagle-Picher Research Lab (West)  
200 Ninth Avenue, N.E.  
Miami, OK 74354  
918/540-1507 800/331-3144

Robert Greer, Repository Manager

### Cooler Returns:

T. Head and Company  
950 Herndon Parkway  
Suite 230  
Herndon, VA 22070  
703/478-3886

John Carria  
Johnetta Sowell

### ERT Edison:

USEPA Environ. Response Branch  
GSA Raritan Depot  
Woodbridge Avenue  
Edison, NJ 08837  
FTS 340-6649, 6689, 6743

Royal Nadeau  
George Prince

**CONTRACT LABORATORY PROGRAM  
REGIONAL DEPUTY PROJECT OFFICERS**

Deb Szaro	USEPA Region I, ESD 60 Westview Street Lexington, MA 02173	617/860-4312
Lou Bevilacqua	USEPA Region II, ESD Woodbridge Avenue Building 209 Edison, NJ 08837	201/321-6702 FTS 340-6702
Chuck Sands	USEPA Region III, CRL 839 Bestgate Road Annapolis, MD 21401	301/266-9180
Tom B. Bennett, Jr.	USEPA Region IV, ESD (ASB) Analytical Support Branch College Station Road Athens, GA 30613	404/546-3112 FTS 250-3112
Pat Churilla	USEPA Region V, ESD 536 S. Clark Street Tenth Floor, CRL Chicago, IL 60605	312/353-9087 FTS 353-9087
David Stockton	USEPA Region VI Laboratory Monterey Park Pl. Bldg. C 6608 Hornwood Drive Houston, TX 77074	713/953-3425 FTS 526-9425
Debra Morey	USEPA Region VII, ESD 25 Funston Road Kansas City, KS 66115	913/236-3881 FTS 757-3881
Eva Hoffman	USEPA Region VIII Laboratory Denver Federal Center Box 25366 Lakewood, CO 80225	303/236-7371 FTS 776-7371
Kent Kitchingman	USEPA Region IX, OPM 215 Fremont Street San Francisco, CA 94105	415/974-0924 FTS 454-0924
Gerald Muth	USEPA Region X Laboratory P.O. Box 549 Manchester, WA 98353	206/442-0370 FTS 399-0370

**REGIONAL SAMPLE CONTROL CENTERS**  
**November 1988**

**CLIENT**

**AUTHORIZED REQUESTORS**

**Region I**

Rosalie Baldassari\*  
617/573-5798; FTS 833-1798

Susan Willis  
617/573-5607; FTS 833-5607

**Region II**

Darvene Adams  
201/321-6705; FTS 340-6705

Gayatri Mehta  
201/321-6705; FTS 340-6705

Regina Odubo-Sullivan  
201/321-6705; FTS 340-6705

Sharon Steltz\*  
201/321-6705; FTS 340-6705

**Region III**

John Austin  
301/266-9180

Dan Donnelly  
301/266-9180

Patricia Krantz  
301/266-9180

Annette Lage  
301/266-9180

Colleen Walling\*  
301/266-9180

**Region IV**

Tom B. Bennett, Jr.  
404/546-3112; FTS 250-3112

Debbie Colquitt  
404/546-3388; FTS 250-3388

Doug Lair\*  
404/546-3300; FTS 250-3300

Doug Mundrick  
404/546-3321; FTS 250-3321

Myron Stephenson  
404/546-3385; FTS 250-3385

---

\*Primary Authorized Requestor

**Region V**

Jan Pels\*  
312/353-2720; FTS 353-2720

Curtis Ross  
312/353-8370; FTS 353-8370

**RAS Only**

CH<sub>2</sub>M Hill:

Jeff Keiser  
Dave Shekoski  
Shirley Stringer  
414/272-2426

CDM:

Cynthia Clark  
Wendy Dewar  
Jill Line  
312/786-1313

E & E:

Renee Hix-Mays  
312/663-9415

Michigan DNR:

Denise Grubin  
517/373-4825

Minnesota PCA:

Dave Kouloski  
Becky Lofgrim  
612/296-7735

Weston:

Eileen Helmer  
Maureen O'Mara  
Melodie Sullivan  
312/993-1067

Wisconsin DNR:

Maureen McCurdy  
Kim McCutchen  
608/267-5063

**Region VI**

Myra Perez\*  
713/953-3425; FTS 526-9425

Hank Thompson  
214/665-6491; FTS 255-6491

**RAS Only**

E & E

John Totin  
214/742-6601

---

\*Primary Authorized Requestor

**Region VII**

Bill Bunn  
913/236-3881; FTS 757-3881

Joyce W. Casper\*  
913/236-3881; FTS 757-3881

Debra Morey  
913/236-3881; FTS 757-3881

**Region VIII**

Barbara Daboll\*  
303/236-7370; FTS 776-7370

RAS Only

CCJM:

Bill Berning  
Jerilyn Guthrie  
303/433-6966

CH<sub>2</sub>M Hill:

Dewey Brigham  
Jane Grogan  
303/771-0900

E & E

Lynn Fischer  
Steve Ignelzi  
303/757-4984

Jacobs:

Pam McDevitt  
Joyce Miyagishima  
303/232-7093

**Region IX**

Thomas Huetteman\*  
415/974-0923; FTS 454-0923

Denise Toll  
415/974-8004; FTS 454-8004

**Region X**

Joyce Crosson  
206/442-2111; FTS 399-2111

Kelsey Ramey  
206/442-4323; FTS 399-4323

Rhonda Wregglesworth\*  
206/442-7121; FTS 399-7121

---

\*Primary Authorized Requestor

## **APPENDIX C**

### **RAS DELIVERABLES AND DATA REPORTING FORMS**

**RAS Organics  
Delivery Requirements**

**A. Contract Start-Up Plan**

The contract laboratory must submit a start-up plan for PO approval that details the laboratory's proposed schedule for receiving samples. The laboratory will be required to receive samples within thirty days of contract award.

**B. Updated Standard Operating Procedures**

The contract laboratory must submit updated copies of all required SOPs that were submitted prior to contract award. The updated SOPs must address all issues of laboratory performance and operation identified during the preaward evaluation process.

**C. Sample Traffic Reports**

The original Sample TR must be returned to SMO with laboratory receipt information for each sample in the SDG. TRs must be submitted in SDG sets with an SDG coversheet attached.

**D. Sample Data Summary Package**

A sample data summary package must be delivered to SMO with other required sample data. The sample data summary package consists of copies of specified items from the sample data package. The sample data summary package must contain data for samples in an SDG, as follows:

1. Case Narrative
2. By fraction (VOA, SV, PEST) and by sample within each fraction - tabulated target compound results (Form I) and tentatively identified compounds (Form I, TIC)(VOA and SV only)
3. By fraction (VOA, SV, PEST) - surrogate spike analysis results (Form II) by matrix (water and/or soil) and for soil, by concentration (low or medium)
4. By fraction (VOA, SV, PEST) - matrix spike/matrix spike duplicate results (Form III)
5. By fraction (VOA, SV, PEST) - blank data (Form IV) and tabulated results (Form I) including tentatively identified compounds (Form I, TIC)(VOA and SV only)
6. By fraction (VOA, SV only) - internal standard area data (Form VIII)

**E. Sample Data Package**

The sample data package is divided into the five major units described below. The last three units are each specific to an analytical fraction (volatiles, semivolatiles, pesticides/PCBs). The sample data package must include data for analyses of all samples in one SDG, including field samples, reanalyses, blanks, matrix spikes, and matrix spike duplicates. The sample data package must include the following:

1. Case Narrative

The Case Narrative must contain: laboratory name; Case number; sample numbers in the SDG, differentiating between initial analyses and reanalyses; SDG number; contract number; and detailed documentation of any quality control, sample, shipment and/or analytical problems encountered in processing the samples reported in the data package.

2. Traffic Reports

A copy of the Sample TRs in Item C must be submitted for all of the samples in an SDG. The TRs must be arranged in increasing EPA sample number order.

3. Volatiles Data

a. QC Summary

(1) Surrogate Percent Recovery Summary (Form II VOA)

(2) Matrix Spike/Matrix Spike Duplicate Summary (Form III VOA)

(3) Method Blank Summary

(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)

(4) GC/MS Tuning and Mass Calibration (Form V VOA)  
BFB in chronological order; by instrument

(5) Internal Standard Area Summary (Form VIII VOA)  
In chronological order; by instrument

b. Sample Data

Sample data must be arranged in packets with the Organic Analysis Data Sheet (Form I VOA, including Form I VOA-TIC) followed by the raw data. Sample packets should be placed in increasing EPA sample number order.

(1) TCL Results - Organic Analysis Data Sheet (Form I VOA)

(2) Tentatively Identified Compounds (Form I VOA-TIC)

This form must be included even if no compounds are found. If so, indicate this on the form by entering "0" in the field for "Number found."

(3) Reconstructed total ion chromatograms (RIC) for each sample or sample extract

(4) For each sample, by each compound identified:

(a) Copies of raw spectra and copies of background-subtracted mass spectra of target compounds



- (b) Copies of mass spectra of Tentatively Identified Compounds with associated best-match spectra (three best matches)
- c. Standards Data
  - (1) Initial Calibration Data (Form VI VOA) - in order by instrument, if more than one instrument used
    - (a) VOA standard(s) reconstructed ion chromatograms and quantitation reports for the initial calibration. Spectra are not required.
    - (b) All initial calibration data must be included, regardless of when it was performed and for which case. When more than one initial calibration is performed, the data must be put in chronological order, by instrument.
  - (2) Continuing Calibration (Form VII VOA) - in order by instrument, if more than one instrument used
    - (a) VOA standard(s) reconstructed ion chromatograms and quantitation reports for all continuing calibrations. Spectra are not required.
    - (b) When more than one continuing calibration is performed, forms must be in chronological order, within fraction and instrument.
  - (3) Internal Standard Area Summary (Form VIII VOA) - in order by instrument, if more than one instrument used
 

When more than one continuing calibration is performed, forms must be in chronological order, by instrument.
- d. Raw QC Data
  - (1) BFB for each GC/MS system utilized
    - (a) Bar graph spectrum
    - (b) Mass listing
  - (2) Blank Data - in chronological order
    - (a) Tabulated results (Form I VOA)
    - (b) Tentatively Identified Compounds (Form I VOA-TIC) even if none found
    - (c) Reconstructed ion chromatogram(s) and quantitation report(s) (GC/MS)
    - (d) TCL spectra with lab generated standard. Data systems which are incapable of dual display must provide spectra in order:

- o Raw TCL compound spectra
- o Enhanced or background subtracted spectra
- o Laboratory generated TCL standard spectra
- (e) GC/MS library search spectra for Tentatively Identified Compounds.
- (f) Quantitation/Calculation of Tentatively Identified Compounds.
- (3) Matrix Spike Data
  - (a) Tabulated results (Form I VOA) of nonspiked TCL compounds. Form I VOA-TIC is not required.
  - (b) Reconstructed ion chromatogram(s) and quantitation report(s) (GC/MS). Spectra are not required.
- (4) Matrix Spike Duplicate Data
  - (a) Tabulated results (Form I VOA) of nonspiked TCL compounds. Form I VOA-TIC is not required.
  - (b) Reconstructed ion chromatogram(s) and quantitation report(s) (GC/MS). Spectra are not required.
  - (c) TCL spectra with lab generated standard. Data systems which are incapable of dual display must provide spectra in order:
    - o Raw TCL compound spectra
    - o Enhanced or background subtracted spectra
    - o Laboratory generated TCL standard spectra
  - (d) GC/MS library search spectra for Tentatively Identified Compounds.
  - (e) Quantitation/Calculation of Tentatively Identified Compounds.
- (3) Matrix Spike Data
  - (a) Tabulated results (Form I) of nonspiked TCL compounds. Form I SV-TIC is not required.
  - (b) Reconstructed ion chromatogram(s) and quantitation report(s) (GC/MS). Spectra are not required.

#### 4. Semivolatiles Data

##### a. QC Summary

- (1) Surrogate Percent Recovery Summary (Form II SV)
- (2) Matrix Spike/Matrix Spike Duplicate Summary (Form III SV)

- (3) Method Blank Summary (Form IV SV)  
(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)
- (4) GC/MS Tuning and Mass Calibration (Form V SV)  
DFTPP in chronological order; by instrument
- (5) Internal Standard Area Summary (Form VIII SV)  
In chronological order; by instrument

b. Sample Data

Sample data must be arranged in packets with the Organic Analysis Data Sheet (Form I SV, including Form I SV-TIC) followed by the raw data. Sample packets should be placed in increasing EPA sample number order.

- (1) TCL Results - Organic Analysis Data Sheet (Form I SV-1, SV-2)
- (2) Tentatively Identified Compounds (Form I SV-TIC)  
This form must be included even if no compounds are found. If so, indicate this on the form by entering "0" in the field for "Number found".
- (3) Reconstructed total ion chromatograms (RIC) for each sample, sample extract, standard, blank, and spiked sample
- (4) For each sample, by each compound identified:
  - (a) Copies of raw spectra and copies of background-subtracted mass spectra of target compounds
  - (b) Copies of mass spectra of Tentatively Identified Compounds with associated best-match spectra (three best matches)
  - (c) GPC chromatograms (if GPC performed)

c. Standards Data

- (1) Initial Calibration Data (Form VI SV-1, SV-2) - in order by instrument, if more than one instrument used
  - (a) BNA standard(s) reconstructed ion chromatograms and quantitation reports for the initial calibration. Spectra are not required.
  - (b) All initial calibration data must be included, regardless of when it was performed and for which case. When more than one initial calibration is performed, the data must be put in chronological order, by instrument.

- (2) Continuing Calibration (Form VII SV-1, SV-2) - in order by instrument, if more than one instrument used
  - (a) BNA standard(s) reconstructed ion chromatograms and quantitation reports for all continuing calibrations. Spectra are not required.
  - (b) When more than one continuing calibration is performed, forms must be in chronological order, by instrument.
- (3) Internal Standard Area Summary (Form VIII SV-1, SV-2) - in order by instrument, if more than one instrument used

When more than one continuing calibration is performed, forms must be in chronological order by instrument.

d. Raw QC Data

- (1) DFTPP for each GC/MS system utilized
  - (a) Bar graph spectrum
  - (b) Mass listing
- (2) Blank Data - in chronological order
  - (a) Tabulated results (Form I SV-1, SV-2)
  - (b) Tentatively Identified Compounds (Form I SV-TIC) - even if none found

5. Pesticide/PCB Data

a. QC Summary

- (1) Surrogate Percent Recovery Summary (Form II PEST)
- (2) Matrix Spike/Matrix Spike Duplicate Summary (Form III PEST)
- (3) Method Blank Summary (Form IV PEST)

(If more than a single form is necessary, forms must be arranged in chronological order by date of analysis of the blank.)

b. Sample Data

Sample data must be arranged in packets with the Organic Analysis Data Sheet (Form I PEST) followed by the raw data. Sample packets should be placed in increasing EPA sample number order.

- (1) TCL Results - Organic Analysis Data Sheet (Form I PEST)
- (2) Copies of pesticide chromatograms

- (3) Copies of pesticide chromatograms from second GC column confirmation
- (4) GC Integration report or data system printout and calibration plots (area vs. concentration) for 4,4'-DDT, 4,4'-DDD, 4,4'-DDE or toxaphene (where appropriate)
- (5) Manual work sheets
- (6) UV traces from GPC (if available)
- (7) Copies of raw spectra and copies of background-subtracted mass spectra of target compounds (if pesticide/PCBs are confirmed by GC/MS)

c. Standards Data

- (1) Form VIII PEST - Pesticide Evaluation Standards Summary (all GC columns)
- (2) Form IX PEST - Pesticide/PCB Standards Summary (all GC columns)
- (3) Form X PEST - Pesticide/PCB Identification (only required for positive results)
- (4) Pesticide standard chromatograms and data system printouts for all standards

d. Raw QC Data

- (1) Blank Data - in chronological order
  - (a) Tabulated results (Form I PEST)
  - (b) Chromatogram(s) and data system printout(s) (GC) for each GC column and instrument used for analysis
- (2) Matrix Spike Data
  - (a) Tabulated results (Form I PEST) of nonspike TCL compounds
  - (b) Chromatogram(s) and data system printout(s) (GC)
- (3) Matrix Spike Duplicate Data
  - (a) Tabulated results (Form I PEST) of nonspike TCL compounds
  - (b) Chromatogram(s) and data system printout(s) (GC)

F. Data in Computer-Readable Form

The contract laboratory must provide a computer-readable copy of the data on data reporting Forms I-X for all samples in an SDG. Computer-readable data deliverables must be submitted on IBM or IBM-compatible, 5.25 inch floppy double-sided, double

density 360 K-byte or a high density 1.2 M-byte diskette. The data must be recorded in ASCII text file format and must adhere to the file, record and field specifications listed in the SOW.

G. GC/MS Tapes

The contract laboratory must store all raw and processed GC/MS data on magnetic tape, in appropriate instrument manufacturer's format. This tape must include data for samples, blanks, matrix spikes, matrix spike duplicates, initial calibrations, continuing calibrations, BFB and DFTPP, as well as all laboratory-generated spectral libraries and quantitation reports required to generate the data package. The Contractor must maintain a written reference logbook of tape files to EPA sample number, calibration data, standards, blanks, matrix spikes, and matrix spike duplicates.

H. Extracts

The contract laboratory is required to retain extracts, preserved at 4°C ( $\pm 2^\circ\text{C}$ ), for 365 days following data submission. A logbook of stored extracts must be maintained, listing EPA sample numbers and associated Case and SDG numbers.

I. Complete Case File Purge

The complete case file purge includes all laboratory records received or generated for a specific Case that have not been previously submitted to EPA as a deliverable. These items include but are not limited to: sample tags, custody records, sample tracking records, analysts logbook pages, bench sheets, chromatographic charts, computer printouts, raw data summaries, instrument logbook pages, correspondence, and the document inventory.

**RAS ORGANICS  
DATA REPORTING FORMS**

1A  
VOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix: (soil/water) \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_

Sample wt/vol: \_\_\_\_\_ (g/mL) \_\_\_\_\_ Lab File ID: \_\_\_\_\_

Level: (low/med) \_\_\_\_\_ Date Received: \_\_\_\_\_

% Moisture: not dec. \_\_\_\_\_ Date Analyzed: \_\_\_\_\_

Column: (pack/cap) \_\_\_\_\_ Dilution Factor: \_\_\_\_\_

CONCENTRATION UNITS:  
(ug/L or ug/Kg) \_\_\_\_\_

CAS NO.	COMPOUND	Q
74-87-3	Chloromethane	
74-83-9	Bromomethane	
75-01-4	Vinyl Chloride	
75-00-3	Chloroethane	
75-09-2	Methylene Chloride	
67-64-1	Acetone	
75-15-0	Carbon Disulfide	
75-35-4	1,1-Dichloroethene	
75-34-3	1,1-Dichloroethane	
540-59-0	1,2-Dichloroethene (total)	
67-66-3	Chloroform	
107-06-2	1,2-Dichloroethane	
78-93-3	2-Butanone	
71-55-6	1,1,1-Trichloroethane	
56-23-5	Carbon Tetrachloride	
108-05-4	Vinyl Acetate	
75-27-4	Bromodichloromethane	
78-87-5	1,2-Dichloropropane	
10061-01-5	cis-1,3-Dichloropropene	
79-01-6	Trichloroethene	
124-48-1	Dibromochloromethane	
79-00-5	1,1,2-Trichloroethane	
71-43-2	Benzene	
10061-02-6	trans-1,3-Dichloropropene	
75-25-2	Bromoform	
108-10-1	4-Methyl-2-Pentanone	
591-78-6	2-Hexanone	
127-18-4	Tetrachloroethene	
79-34-5	1,1,2,2-Tetrachloroethane	
108-88-3	Toluene	
108-90-7	Chlorobenzene	
100-41-4	Ethylbenzene	
100-42-5	Styrene	
1330-20-7	Xylene (total)	



1B  
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix: (soil/water) \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_

Sample wt/vol: \_\_\_\_\_ (g/mL) \_\_\_\_\_ Lab File ID: \_\_\_\_\_

Level: (low/med) \_\_\_\_\_ Date Received: \_\_\_\_\_

% Moisture: not dec. \_\_\_\_\_ dec. \_\_\_\_\_ Date Extracted: \_\_\_\_\_

Extraction: (SepF/Cont/Sonc) \_\_\_\_\_ Date Analyzed: \_\_\_\_\_

GPC Cleanup: (Y/N) \_\_\_\_\_ pH: \_\_\_\_\_ Dilution Factor: \_\_\_\_\_

CAS NO.                      COMPOUND                      CONCENTRATION UNITS:  
(ug/L or ug/Kg) \_\_\_\_\_ Q

108-95-2-----	Phenol		
111-44-4-----	bis(2-Chloroethyl) ether		
95-57-8-----	2-Chlorophenol		
541-73-1-----	1,3-Dichlorobenzene		
106-46-7-----	1,4-Dichlorobenzene		
100-51-6-----	Benzyl alcohol		
95-50-1-----	1,2-Dichlorobenzene		
95-48-7-----	2-Methylphenol		
108-60-1-----	bis(2-Chloroisopropyl) ether		
106-44-5-----	4-Methylphenol		
621-64-7-----	N-Nitroso-di-n-propylamine		
67-72-1-----	Hexachloroethane		
98-95-3-----	Nitrobenzene		
78-59-1-----	Isophorone		
88-75-5-----	2-Nitrophenol		
105-67-9-----	2,4-Dimethylphenol		
65-85-0-----	Benzoic acid		
111-91-1-----	bis(2-Chloroethoxy) methane		
120-83-2-----	2,4-Dichlorophenol		
120-82-1-----	1,2,4-Trichlorobenzene		
91-20-3-----	Naphthalene		
106-47-8-----	4-Chloroaniline		
87-68-3-----	Hexachlorobutadiene		
59-50-7-----	4-Chloro-3-methylphenol		
91-57-6-----	2-Methylnaphthalene		
77-47-4-----	Hexachlorocyclopentadiene		
88-06-2-----	2,4,6-Trichlorophenol		
95-95-4-----	2,4,5-Trichlorophenol		
91-58-7-----	2-Chloronaphthalene		
88-74-4-----	2-Nitroaniline		
131-11-3-----	Dimethylphthalate		
208-96-8-----	Acenaphthylene		
606-20-2-----	2,6-Dinitrotoluene		

1C  
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix: (soil/water) \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_

Sample wt/vol: \_\_\_\_\_ (g/mL) \_\_\_\_\_ Lab File ID: \_\_\_\_\_

Level: (low/med) \_\_\_\_\_ Date Received: \_\_\_\_\_

% Moisture: not dec. \_\_\_\_\_ dec. \_\_\_\_\_ Date Extracted: \_\_\_\_\_

Extraction: (SepF/Cont/Sonc) \_\_\_\_\_ Date Analyzed: \_\_\_\_\_

GPC Cleanup: (Y/N) \_\_\_\_\_ pH: \_\_\_\_\_ Dilution Factor: \_\_\_\_\_

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) _____	Q
99-09-2-----	3-Nitroaniline		
83-32-9-----	Acenaphthene		
51-28-5-----	2,4-Dinitrophenol		
100-02-7-----	4-Nitrophenol		
132-64-9-----	Dibenzofuran		
121-14-2-----	2,4-Dinitrotoluene		
84-66-2-----	Diethylphthalate		
7005-72-3-----	4-Chlorophenyl-phenylether		
86-73-7-----	Fluorene		
100-01-6-----	4-Nitroaniline		
534-52-1-----	4,6-Dinitro-2-methylphenol		
86-30-6-----	N-Nitrosodiphenylamine (1)		
101-55-3-----	4-Bromophenyl-phenylether		
118-74-1-----	Hexachlorobenzene		
87-86-5-----	Pentachlorophenol		
85-01-8-----	Phenanthrene		
120-12-7-----	Anthracene		
84-74-2-----	Di-n-butylphthalate		
206-44-0-----	Fluoranthene		
129-00-0-----	Pyrene		
85-68-7-----	Butylbenzylphthalate		
91-94-1-----	3,3'-Dichlorobenzidine		
56-55-3-----	Benzo(a)anthracene		
218-01-9-----	Chrysene		
117-81-7-----	bis(2-Ethylhexyl)phthalate		
117-84-0-----	Di-n-octylphthalate		
205-99-2-----	Benzo(b)fluoranthene		
207-08-9-----	Benzo(k)fluoranthene		
50-32-8-----	Benzo(a)pyrene		
193-39-5-----	Indeno(1,2,3-cd)pyrene		
53-70-3-----	Dibenz(a,h)anthracene		
191-24-2-----	Benzo(g,h,i)perylene		

(1) - Cannot be separated from Diphenylamine

1D  
PESTICIDE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix: (soil/water) \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_

Sample wt/vol: \_\_\_\_\_ (g/mL) \_\_\_\_\_ Lab File ID: \_\_\_\_\_

Level: (low/med) \_\_\_\_\_ Date Received: \_\_\_\_\_

% Moisture: not dec. \_\_\_\_\_ dec. \_\_\_\_\_ Date Extracted: \_\_\_\_\_

Extraction: (SepF/Cont/Sonc) \_\_\_\_\_ Date Analyzed: \_\_\_\_\_

GPC Cleanup: (Y/N) \_\_\_\_\_ pH: \_\_\_\_\_ Dilution Factor: \_\_\_\_\_

CAS NO.	COMPOUND	CONCENTRATION UNITS:	
		(ug/L or ug/Kg) _____	Q
319-84-6-----	alpha-BHC		
319-85-7-----	beta-BHC		
319-86-8-----	delta-BHC		
58-89-9-----	gamma-BHC (Lindane)		
76-44-8-----	Heptachlor		
309-00-2-----	Aldrin		
1024-57-3-----	Heptachlor epoxide		
959-98-8-----	Endosulfan I		
60-57-1-----	Dieldrin		
72-55-9-----	4,4'-DDE		
72-20-8-----	Endrin		
33213-65-9-----	Endosulfan II		
72-54-8-----	4,4'-DDD		
1031-07-8-----	Endosulfan sulfate		
50-29-3-----	4,4'-DDT		
72-43-5-----	Methoxychlor		
53494-70-5-----	Endrin ketone		
5103-71-9-----	alpha-Chlordane		
5103-74-2-----	gamma-Chlordane		
8001-35-2-----	Toxaphene		
12674-11-2-----	Aroclor-1016		
11104-28-2-----	Aroclor-1221		
11141-16-5-----	Aroclor-1232		
53469-21-9-----	Aroclor-1242		
12672-29-6-----	Aroclor-1248		
11097-69-1-----	Aroclor-1254		
11096-82-5-----	Aroclor-1260		

1E  
VOLATILE ORGANICS ANALYSIS DATA SHEET  
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix: (soil/water) \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_

Sample wt/vol: \_\_\_\_\_ (g/mL) \_\_\_\_\_ Lab File ID: \_\_\_\_\_

Level: (low/med) \_\_\_\_\_ Date Received: \_\_\_\_\_

% Moisture: not dec. \_\_\_\_\_ Date Analyzed: \_\_\_\_\_

Column: (pack/cap) \_\_\_\_\_ Dilution Factor: \_\_\_\_\_

Number TICs found: \_\_\_\_\_ CONCENTRATION UNITS:  
(ug/L or ug/Kg) \_\_\_\_\_

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
19.				
20.				
21.				
22.				
23.				
24.				
25.				
26.				
27.				
28.				
29.				
30.				

1F  
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET  
TENTATIVELY IDENTIFIED COMPOUNDS

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix: (soil/water) \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_

Sample wt/vol: \_\_\_\_\_ (g/mL) \_\_\_\_\_ Lab File ID: \_\_\_\_\_

Level: (low/med) \_\_\_\_\_ Date Received: \_\_\_\_\_

% Moisture: not dec. \_\_\_\_\_ dec. \_\_\_\_\_ Date Extracted: \_\_\_\_\_

Extraction: (SepF/Cont/Sonc) \_\_\_\_\_ Date Analyzed: \_\_\_\_\_

GPC Cleanup: (Y/N) \_\_\_\_\_ pH: \_\_\_\_\_ Dilution Factor: \_\_\_\_\_

Number TICs found: \_\_\_\_\_ CONCENTRATION UNITS:  
(ug/L or ug/Kg) \_\_\_\_\_

CAS NUMBER	COMPOUND NAME	RT	EST. CONC.	Q
1.				
2.				
3.				
4.				
5.				
6.				
7.				
8.				
9.				
10.				
11.				
12.				
13.				
14.				
15.				
16.				
17.				
18.				
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20.				
21.				
22.				
23.				
24.				
25.				
26.				
27.				
28.				
29.				
30.				

2A  
WATER VOLATILE SURROGATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

	EPA SAMPLE NO.	S1 (TOL) #	S2 (BFB) #	S3 (DCE) #	OTHER	TOT OUT
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						

QC LIMITS

S1 (TOL) = Toluene-d8 (88-110)  
 S2 (BFB) = Bromofluorobenzene (86-115)  
 S3 (DCE) = 1,2-Dichloroethane-d4 (76-114)

# Column to be used to flag recovery values

\* Values outside of contract required QC limits

D Surrogates diluted out

2B  
SOIL VOLATILE SURROGATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Level: (low/med) \_\_\_\_\_

	EPA SAMPLE NO.	S1 (TOL) #	S2 (BFB) #	S3 (DCE) #	OTHER	TOT OUT
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						

QC LIMITS

S1 (TOL) = Toluene-d8 (81-117)  
 S2 (BFB) = Bromofluorobenzene (74-121)  
 S3 (DCE) = 1,2-Dichloroethane-d4 (70-121)

# Column to be used to flag recovery values

\* Values outside of contract required QC limits

D Surrogates diluted out

2C  
WATER SEMIVOLATILE SURROGATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

	EPA SAMPLE NO.	S1 (NBZ) #	S2 (FBP) #	S3 (TPH) #	S4 (PHL) #	S5 (2FP) #	S6 (TBP) #	OTHER	TOT OUT
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

	QC LIMITS
S1 (NBZ) = Nitrobenzene-d5	(35-114)
S2 (FBP) = 2-Fluorobiphenyl	(43-116)
S3 (TPH) = Terphenyl-d14	(33-141)
S4 (PHL) = Phenol-d5	(10-94)
S5 (2FP) = 2-Fluorophenol	(21-100)
S6 (TBP) = 2,4,6-Tribromophenol	(10-123)

# Column to be used to flag recovery values  
 \* Values outside of contract required QC limits  
 D Surrogates diluted out



2D  
SOIL SEMIVOLATILE SURROGATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Level: (low/med) \_\_\_\_\_

	EPA SAMPLE NO.	S1 (NBZ) #	S2 (FBP) #	S3 (TPH) #	S4 (PHL) #	S5 (2FP) #	S6 (TBP) #	OTHER	TOT OUT
01									
02									
03									
04									
05									
06									
07									
08									
09									
10									
11									
12									
13									
14									
15									
16									
17									
18									
19									
20									
21									
22									
23									
24									
25									
26									
27									
28									
29									
30									

	QC LIMITS
S1 (NBZ) = Nitrobenzene-d5	(35-114)
S2 (FBP) = 2-Fluorobiphenyl	(43-116)
S3 (TPH) = Terphenyl-d14	(33-141)
S4 (PHL) = Phenol-d5	(10-94)
S5 (2FP) = 2-Fluorophenol	(21-100)
S6 (TBP) = 2,4,6-Tribromophenol	(10-123)

# Column to be used to flag recovery values  
 \* Values outside of contract required QC limits  
 D Surrogates diluted out

2E  
WATER PESTICIDE SURROGATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

	EPA SAMPLE NO.	S1 (DBC) #	OTHER
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			

ADVISORY  
QC LIMITS  
(24-154)

S1 (DBC) = Dibutylchloroendate

# Column to be used to flag recovery values

\* Values outside of QC limits

D Surrogates diluted out

2F  
SOIL PESTICIDE SURROGATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Level: (low/med) \_\_\_\_\_

	EPA SAMPLE NO.	S1 (DBC) #	OTHER
01			
02			
03			
04			
05			
06			
07			
08			
09			
10			
11			
12			
13			
14			
15			
16			
17			
18			
19			
20			
21			
22			
23			
24			
25			
26			
27			
28			
29			
30			

ADVISORY  
QC LIMITS  
(20-150)

S1 (DBC) = Dibutylchloroendate

# Column to be used to flag recovery values

\* Values outside of QC limits

D Surrogates diluted out

3A  
WATER VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix Spike - EPA Sample No.: \_\_\_\_\_

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC. LIMITS REC.
1,1-Dichloroethene					61-145
Trichloroethene					71-120
Benzene					76-127
Toluene					76-125
Chlorobenzene					75-130

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LIMITS RPD	REC.
1,1-Dichloroethene					14	61-145
Trichloroethene					14	71-120
Benzene					11	76-127
Toluene					13	76-125
Chlorobenzene					13	75-130

# Column to be used to flag recovery and RPD values with an asterisk

\* Values outside of QC limits

RPD: \_\_\_\_\_ out of \_\_\_\_\_ outside limits

Spike Recovery: \_\_\_\_\_ out of \_\_\_\_\_ outside limits

COMMENTS: \_\_\_\_\_  
\_\_\_\_\_

3B  
SOIL VOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix Spike - EPA Sample No.: \_\_\_\_\_ Level: (low/med) \_\_\_\_\_

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC. LIMITS REC.
1,1-Dichloroethene					59-172
Trichloroethene					62-137
Benzene					66-142
Toluene					59-139
Chlorobenzene					60-133

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	REC.
1,1-Dichloroethene					22	59-172
Trichloroethene					23	62-137
Benzene					21	66-142
Toluene					21	59-139
Chlorobenzene					21	60-133

# Column to be used to flag recovery and RPD values with an asterisk

\* Values outside of QC limits

RPD: \_\_\_\_\_ out of \_\_\_\_\_ outside limits

Spike Recovery: \_\_\_\_\_ out of \_\_\_\_\_ outside limits

COMMENTS: \_\_\_\_\_  
\_\_\_\_\_

3C  
WATER SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Matrix Spike - EPA Sample No.: \_\_\_\_\_

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC LIMITS REC.
Phenol					12- 89
2-Chlorophenol					27-123
1,4-Dichlorobenzene					36- 97
N-Nitroso-di-n-prop. (1)					41-116
1,2,4-Trichlorobenzene					39- 98
4-Chloro-3-methylphenol					23- 97
Acenaphthene					46-118
4-Nitrophenol					10- 80
2,4-Dinitrotoluene					24- 96
Pentachlorophenol					9-103
Pyrene					26-127

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LIMITS RPD REC.
Phenol					42 12- 89
2-Chlorophenol					40 27-123
1,4-Dichlorobenzene					28 36- 97
N-Nitroso-di-n-prop. (1)					38 41-116
1,2,4-Trichlorobenzene					28 39- 98
4-Chloro-3-methylphenol					42 23- 97
Acenaphthene					31 46-118
4-Nitrophenol					50 10- 80
2,4-Dinitrotoluene					38 24- 96
Pentachlorophenol					50 9-103
Pyrene					31 26-127

(1) N-Nitroso-di-n-propylamine

# Column to be used to flag recovery and RPD values with an asterisk  
 \* Values outside of QC limits

RPD: \_\_\_\_\_ out of \_\_\_\_\_ outside limits  
 Spike Recovery: \_\_\_\_\_ out of \_\_\_\_\_ outside limits

COMMENTS: \_\_\_\_\_  
 \_\_\_\_\_

3D  
SOIL SEMIVOLATILE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Matrix Spike - EPA Sample No.: \_\_\_\_\_ Level: (low/med) \_\_\_\_\_

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
Phenol					26- 90
2-Chlorophenol					25-102
1,4-Dichlorobenzene					28-104
N-Nitroso-di-n-prop. (1)					41-126
1,2,4-Trichlorobenzene					38-107
4-Chloro-3-methylphenol					26-103
Acenaphthene					31-137
4-Nitrophenol					11-114
2,4-Dinitrotoluene					28- 89
Pentachlorophenol					17-109
Pyrene					35-142

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	REC.
Phenol					35	26- 90
2-Chlorophenol					50	25-102
1,4-Dichlorobenzene					27	28-104
N-Nitroso-di-n-prop. (1)					38	41-126
1,2,4-Trichlorobenzene					23	38-107
4-Chloro-3-methylphenol					33	26-103
Acenaphthene					19	31-137
4-Nitrophenol					50	11-114
2,4-Dinitrotoluene					47	28- 89
Pentachlorophenol					47	17-109
Pyrene					36	35-142

(1) N-Nitroso-di-n-propylamine

# Column to be used to flag recovery and RPD values with an asterisk  
 \* Values outside of QC limits

RPD: \_\_\_\_\_ out of \_\_\_\_\_ outside limits  
 Spike Recovery: \_\_\_\_\_ out of \_\_\_\_\_ outside limits

COMMENTS: \_\_\_\_\_  
 \_\_\_\_\_

3E  
WATER PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix Spike - EPA Sample No.: \_\_\_\_\_

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	MS CONCENTRATION (ug/L)	MS % REC #	QC. LIMITS REC.
gamma-BHC (Lindane)					56-123
Heptachlor					40-131
Aldrin					40-120
Dieldrin					52-126
Endrin					56-121
4,4'-DDT					38-127

COMPOUND	SPIKE ADDED (ug/L)	MSD CONCENTRATION (ug/L)	MSD % REC #	% RPD #	QC LIMITS RPD	REC.
gamma-BHC (Lindane)					15	56-123
Heptachlor					20	40-131
Aldrin					22	40-120
Dieldrin					18	52-126
Endrin					21	56-121
4,4'-DDT					27	38-127

# Column to be used to flag recovery and RPD values with an asterisk

\* Values outside of QC limits

RPD: \_\_\_\_\_ out of \_\_\_\_\_ outside limits

Spike Recovery: \_\_\_\_\_ out of \_\_\_\_\_ outside limits

COMMENTS: \_\_\_\_\_  
\_\_\_\_\_



3F  
SOIL PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Matrix Spike - EPA Sample No.: \_\_\_\_\_ Level: (low/med) \_\_\_\_\_

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC. LIMITS REC.
=====	=====	=====	=====	=====	=====
gamma-BHC (Lindane)					46-127
Heptachlor					35-130
Aldrin					34-132
Dieldrin					31-134
Endrin					42-139
4,4'-DDT					23-134

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	REC.
=====	=====	=====	=====	=====	=====	=====
gamma-BHC (Lindane)					50	46-127
Heptachlor					31	35-130
Aldrin					43	34-132
Dieldrin					38	31-134
Endrin					45	42-139
4,4'-DDT					50	23-134

# Column to be used to flag recovery and RPD values with an asterisk

\* Values outside of QC limits

RPD: \_\_\_\_\_ out of \_\_\_\_\_ outside limits  
 Spike Recovery: \_\_\_\_\_ out of \_\_\_\_\_ outside limits

COMMENTS: \_\_\_\_\_  
 \_\_\_\_\_

4A  
VOLATILE METHOD BLANK SUMMARY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
Lab File ID: \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_  
Date Analyzed: \_\_\_\_\_ Time Analyzed: \_\_\_\_\_  
Matrix: (soil/water) \_\_\_\_\_ Level: (low/med) \_\_\_\_\_  
Instrument ID: \_\_\_\_\_

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS: \_\_\_\_\_  
\_\_\_\_\_

4B  
SEMIVOLATILE METHOD BLANK SUMMARY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Lab File ID: \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_

Date Extracted: \_\_\_\_\_ Extraction: (SepF/Cont/Sonc) \_\_\_\_\_

Date Analyzed: \_\_\_\_\_ Time Analyzed: \_\_\_\_\_

Matrix: (soil/water) \_\_\_\_\_ Level: (low/med) \_\_\_\_\_

Instrument ID: \_\_\_\_\_

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	TIME ANALYZED
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				
27				
28				
29				
30				

COMMENTS: \_\_\_\_\_

\_\_\_\_\_

4C  
PESTICIDE METHOD BLANK SUMMARY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Lab Sample ID: \_\_\_\_\_ Lab File ID: \_\_\_\_\_

Matrix: (soil/water) \_\_\_\_\_ Level: (low/med) \_\_\_\_\_

Date Extracted: \_\_\_\_\_ Extraction: (SepF/Cont/Sonc) \_\_\_\_\_

Date Analyzed (1): \_\_\_\_\_ Date Analyzed (2): \_\_\_\_\_

Time Analyzed (1): \_\_\_\_\_ Time Analyzed (2): \_\_\_\_\_

Instrument ID (2): \_\_\_\_\_ Instrument ID (2): \_\_\_\_\_

GC Column ID (1): \_\_\_\_\_ GC Column ID (1): \_\_\_\_\_

THIS METHOD BLANK APPLIES TO THE FOLLOWING SAMPLES, MS AND MSD:

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED 1	DATE ANALYZED 2
01				
02				
03				
04				
05				
06				
07				
08				
09				
10				
11				
12				
13				
14				
15				
16				
17				
18				
19				
20				
21				
22				
23				
24				
25				
26				

COMMENTS: \_\_\_\_\_

\_\_\_\_\_

5A  
VOLATILE ORGANIC GC/MS TUNING AND MASS  
CALIBRATION - BROMOFLUOROBENZENE (BFB)

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Lab File ID: \_\_\_\_\_ BFB Injection Date: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ BFB Injection Time: \_\_\_\_\_  
 Matrix: (soil/water) \_\_\_\_\_ Level: (low/med) \_\_\_\_\_ Column: (pack/cap) \_\_\_\_\_

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
50	15.0 - 40.0% of mass 95	
75	30.0 - 60.0% of mass 95	
95	Base peak, 100% relative abundance	
96	5.0 - 9.0% of mass 95	
173	Less than 2.0% of mass 174	( ) 1
174	Greater than 50.0% of mass 95	
175	5.0 - 9.0% of mass 174	( ) 1
176	Greater than 95.0%, but less than 101.0% of mass 174	( ) 1
177	5.0 - 9.0% of mass 176	( ) 2

1-Value is % mass 174

2-Value is % mass 176

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

5B  
SEMIVOLATILE ORGANIC GC/MS TUNING AND MASS  
CALIBRATION - DECAFLUOROTRIPHENYLPHOSPHINE (DFTPP)

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Lab File ID: \_\_\_\_\_ DFTPP Injection Date: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ DFTPP Injection Time: \_\_\_\_\_

m/e	ION ABUNDANCE CRITERIA	% RELATIVE ABUNDANCE
51	30.0 - 60.0% of mass 198	
68	Less than 2.0% of mass 69	( ) 1
69	Mass 69 relative abundance	
70	Less than 2.0% of mass 69	( ) 1
127	40.0 - 60.0% of mass 198	
197	Less than 1.0% of mass 198	
198	Base Peak, 100% relative abundance	
199	5.0 to 9.0% of mass 198	
275	10.0 - 30.0% of mass 198	
365	Greater than 1.00% of mass 198	
441	Present, but less than mass 443	
442	Greater than 40.0% of mass 198	
443	17.0 - 23.0% of mass 442	( ) 2

1-Value is % mass 69

2-Value is % mass 442

THIS TUNE APPLIES TO THE FOLLOWING SAMPLES, MS, MSD, BLANKS, AND STANDARDS:

	EPA SAMPLE NO.	LAB SAMPLE ID	LAB FILE ID	DATE ANALYZED	TIME ANALYZED
01					
02					
03					
04					
05					
06					
07					
08					
09					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					

page \_\_\_ of \_\_\_

FORM V SV

1/87 Rev.

6A  
VOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Instrument ID: \_\_\_\_\_ Calibration Date(s): \_\_\_\_\_

Matrix: (soil/water) \_\_\_\_\_ Level: (low/med) \_\_\_\_\_ Column: (pack/cap) \_\_\_\_\_

Min  $\overline{RRF}$  for SPCC(#) = 0.300 (0.250 for Bromoform) Max %RSD for CCC(\*) = 30.0%

LAB FILE ID: _____	RRF20 = _____	RRF50 = _____
RRF100= _____	RRF150= _____	RRF200= _____

COMPOUND	RRF20	RRF50	RRF100	RRF150	RRF200	$\overline{RRF}$	% RSD
Chloromethane	#						#
Bromomethane							
Vinyl Chloride	*						*
Chloroethane							
Methylene Chloride							
Acetone							
Carbon Disulfide							
1,1-Dichloroethene	*						*
1,1-Dichloroethane	#						#
1,2-Dichloroethene (total)							
Chloroform	*						*
1,2-Dichloroethane							
2-Butanone							
1,1,1-Trichloroethane							
Carbon Tetrachloride							
Vinyl Acetate							
Bromodichloromethane							
1,2-Dichloropropane	*						*
cis-1,3-Dichloropropene							
Trichloroethene							
Dibromochloromethane							
1,1,2-Trichloroethane							
Benzene							
trans-1,3-Dichloropropene							
Bromoform	#						#
4-Methyl-2-Pentanone							
2-Hexanone							
Tetrachloroethene							
1,1,2,2-Tetrachloroethane	#						#
Toluene	*						*
Chlorobenzene	#						#
Ethylbenzene	*						*
Styrene							
Xylene (total)							
Toluene-d8							
Bromofluorobenzene							
1,2-Dichloroethane-d4							

6B  
SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Instrument ID: \_\_\_\_\_ Calibration Date(s): \_\_\_\_\_

Min  $\overline{RRF}$  for SPCC(#) = 0.050

Max %RSD for CCC(\*) = 30.0%

LAB FILE ID: _____	RRF20 = _____	RRF50 = _____					
RRF80 = _____	RRF120 = _____	RRF160 = _____					
COMPOUND	RRF20	RRF50	RRF80	RRF120	RRF160	$\overline{RRF}$	% RSD
Phenol	*						*
bis(2-Chloroethyl) ether							
2-Chlorophenol							
1,3-Dichlorobenzene							
1,4-Dichlorobenzene	*						*
Benzyl alcohol							
1,2-Dichlorobenzene							
2-Methylphenol							
bis(2-Chloroisopropyl) ether							
4-Methylphenol							
N-Nitroso-di-n-propylamine	#						
Hexachloroethane							
Nitrobenzene							
Isophorone							
2-Nitrophenol	*						*
2,4-Dimethylphenol							
Benzoic acid							
bis(2-Chloroethoxy) methane							
2,4-Dichlorophenol	*						*
1,2,4-Trichlorobenzene							
Naphthalene							
4-Chloroaniline							
Hexachlorobutadiene	*						*
4-Chloro-3-methylphenol	*						*
2-Methylnaphthalene							
Hexachlorocyclopentadiene	#						#
2,4,6-Trichlorophenol	*						*
2,4,5-Trichlorophenol							
2-Chloronaphthalene							
2-Nitroaniline							
Dimethylphthalate							
Acenaphthylene							
2,6-Dinitrotoluene							
3-Nitroaniline							
Acenaphthene	*						*
2,4-Dinitrophenol	#						#
4-Nitrophenol	#						#



6C  
SEMIVOLATILE ORGANICS INITIAL CALIBRATION DATA

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Instrument ID: \_\_\_\_\_ Calibration Date(s): \_\_\_\_\_

Min RRF for SPCC(#) = 0.050

Max %RSD for CCC(\*) = 30.0%

LAB FILE ID: _____	RRF20 = _____	RRF50 = _____
RRF80 = _____	RRF120 = _____	RRF160 = _____

COMPOUND	RRF20	RRF50	RRF80	RRF120	RRF160	RRF	% RSD
Dibenzofuran							
2,4-Dinitrotoluene							
Diethylphthalate							
4-Chlorophenyl-phenylether							
Fluorene							
4-Nitroaniline							
4,6-Dinitro-2-methylphenol							
N-Nitrosodiphenylamine (1) *							*
4-Bromophenyl-phenylether							
Hexachlorobenzene							
Pentachlorophenol *							*
Phenanthrene							
Anthracene							
Di-n-butylphthalate							
Fluoranthene *							*
Pyrene							
Butylbenzylphthalate							
3,3'-Dichlorobenzidine							
Benzo(a)anthracene							
Chrysene							
bis(2-Ethylhexyl)phthalate							
Di-n-octylphthalate *							*
Benzo(b)fluoranthene							
Benzo(k)fluoranthene							
Benzo(a)pyrene *							*
Indeno(1,2,3-cd)pyrene							
Dibenz(a,h)anthracene							
Benzo(g,h,i)perylene							
=====							
Nitrobenzene-d5							
2-Fluorobiphenyl							
Terphenyl-d14							
Phenol-d5							
2-Fluorophenol							
2,4,6-Tribromophenol							*

(1) Cannot be separated from Diphenylamine

7A  
VOLATILE CONTINUING CALIBRATION CHECK

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Instrument ID: \_\_\_\_\_ Calibration Date: \_\_\_\_\_ Time: \_\_\_\_\_

Lab File ID: \_\_\_\_\_ Init. Calib. Date(s): \_\_\_\_\_

Matrix: (soil/water) \_\_\_\_\_ Level: (low/med) \_\_\_\_\_ Column: (pack/cap) \_\_\_\_\_

Min RRF50 for SPCC(#) = 0.300 (0.250 for Bromoform) Max %D for CCC(\*) = 25.0%

COMPOUND	RRF	RRF50	%D
Chloromethane	#		#
Bromomethane			
Vinyl Chloride	*		*
Chloroethane			
Methylene Chloride			
Acetone			
Carbon Disulfide			
1,1-Dichloroethene	*		*
1,1-Dichloroethane	#		#
1,2-Dichloroethene (total)			
Chloroform	*		*
1,2-Dichloroethane			
2-Butanone			
1,1,1-Trichloroethane			
Carbon Tetrachloride			
Vinyl Acetate			
Bromodichloromethane			
1,2-Dichloropropane	*		*
cis-1,3-Dichloropropene			
Trichloroethene			
Dibromochloromethane			
1,1,2-Trichloroethane			
Benzene			
trans-1,3-Dichloropropene			
Bromoform	#		#
4-Methyl-2-Pentanone			
2-Hexanone			
Tetrachloroethene			
1,1,2,2-Tetrachloroethane	#		#
Toluene	*		*
Chlorobenzene	#		#
Ethylbenzene	*		*
Styrene			
Xylene (total)			
Toluene-d8			
Bromofluorobenzene			
1,2-Dichloroethane-d4			

7B  
SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Calibration Date: \_\_\_\_\_ Time: \_\_\_\_\_  
 Lab File ID: \_\_\_\_\_ Init. Calib. Date(s): \_\_\_\_\_

Min RRF50 for SPCC(#) = 0.050

Max %D for CCC(\*) = 25.0%

COMPOUND	RRF	RRF50	%D
Phenol	*		*
bis(2-Chloroethyl) ether			
2-Chlorophenol			
1,3-Dichlorobenzene			
1,4-Dichlorobenzene	*		*
Benzyl alcohol			
1,2-Dichlorobenzene			
2-Methylphenol			
bis(2-Chloroisopropyl) ether			
4-Methylphenol			
N-Nitroso-di-n-propylamine	#		
Hexachloroethane			
Nitrobenzene			
Isophorone			
2-Nitrophenol	*		*
2,4-Dimethylphenol			
Benzoic acid			
bis(2-Chloroethoxy) methane			
2,4-Dichlorophenol	*		*
1,2,4-Trichlorobenzene			
Naphthalene			
4-Chloroaniline			
Hexachlorobutadiene	*		*
4-Chloro-3-methylphenol	*		*
2-Methylnaphthalene			
Hexachlorocyclopentadiene	#		#
2,4,6-Trichlorophenol	*		*
2,4,5-Trichlorophenol			
2-Chloronaphthalene			
2-Nitroaniline			
Dimethylphthalate			
Acenaphthylene			
2,6-Dinitrotoluene			
3-Nitroaniline			
Acenaphthene	*		*
2,4-Dinitrophenol	#		#
4-Nitrophenol	#		#

7C  
SEMIVOLATILE CONTINUING CALIBRATION CHECK

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Calibration Date: \_\_\_\_\_ Time: \_\_\_\_\_  
 Lab File ID: \_\_\_\_\_ Init. Calib. Date(s): \_\_\_\_\_  
 Min RRF50 for SPCC(#) = 0.050 Max %D for CCC(\*) = 25.0%

COMPOUND	RRF	RRF50	%D
Dibenzofuran			
2,4-Dinitrotoluene			
Diethylphthalate			
4-Chlorophenyl-phenylether			
Fluorene			
4-Nitroaniline			
4,6-Dinitro-2-methylphenol			
N-Nitrosodiphenylamine (1) *			*
4-Bromophenyl-phenylether			
Hexachlorobenzene			
Pentachlorophenol *			*
Phenanthrene			
Anthracene			
Di-n-butylphthalate			
Fluoranthene *			*
Pyrene			
Butylbenzylphthalate			
3,3'-Dichlorobenzidine			
Benzo(a)anthracene			
Chrysene			
bis(2-Ethylhexyl)phthalate			
Di-n-octylphthalate *			*
Benzo(b)fluoranthene			
Benzo(k)fluoranthene			
Benzo(a)pyrene *			*
Indeno(1,2,3-cd)pyrene			
Dibenz(a,h)anthracene			
Benzo(g,h,i)perylene			
Nitrobenzene-d5			
2-Fluorobiphenyl			
Terphenyl-d14			
Phenol-d5			
2-Fluorophenol			
2,4,6-Tribromophenol			

(1) Cannot be separated from Diphenylamine

8A  
VOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Lab File ID (Standard): \_\_\_\_\_ Date Analyzed: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Time Analyzed: \_\_\_\_\_  
 Matrix: (soil/water) \_\_\_\_\_ Level: (low/med) \_\_\_\_\_ Column: (pack/cap) \_\_\_\_\_

	IS1(BCM)		IS2(DFB)		IS3(CBZ)	
	AREA #	RT	AREA #	RT	AREA #	RT
=====	=====	=====	=====	=====	=====	=====
12 HOUR STD						
=====	=====	=====	=====	=====	=====	=====
UPPER LIMIT						
=====	=====	=====	=====	=====	=====	=====
LOWER LIMIT						
=====	=====	=====	=====	=====	=====	=====
EPA SAMPLE NO.						
=====	=====	=====	=====	=====	=====	=====
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						

IS1 (BCM) = Bromochloromethane  
 IS2 (DFB) = 1,4-Difluorobenzene  
 IS3 (CBZ) = Chlorobenzene

UPPER LIMIT = + 100%  
 of internal standard area.  
 LOWER LIMIT = - 50%  
 of internal standard area.

# Column used to flag internal standard area values with an asterisk

page \_\_ of \_\_

8B  
SEMIVOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Lab File ID (Standard): \_\_\_\_\_ Date Analyzed: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Time Analyzed: \_\_\_\_\_

	IS1 (DCB) AREA #	RT	IS2 (NPT) AREA #	RT	IS3 (ANT) AREA #	RT
=====	=====	=====	=====	=====	=====	=====
12 HOUR STD						
=====	=====	=====	=====	=====	=====	=====
UPPER LIMIT						
=====	=====	=====	=====	=====	=====	=====
LOWER LIMIT						
=====	=====	=====	=====	=====	=====	=====
EPA SAMPLE NO.						
=====	=====	=====	=====	=====	=====	=====
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						

IS1 (DCB) = 1,4-Dichlorobenzene-d4  
 IS2 (NPT) = Naphthalene-d8  
 IS3 (ANT) = Acenaphthene-d8

UPPER LIMIT = + 100%  
 of internal standard area.  
 LOWER LIMIT = - 50%  
 of internal standard area.

# Column used to flag internal standard area values with an asterisk

8C  
SEMIVOLATILE INTERNAL STANDARD AREA SUMMARY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Lab File ID (Standard): \_\_\_\_\_ Date Analyzed: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ Time Analyzed: \_\_\_\_\_

	IS4 (PHN) AREA #	RT	IS5 (CRY) AREA #	RT	IS6 (PRY) AREA #	RT
=====	=====	=====	=====	=====	=====	=====
12 HOUR STD						
=====	=====	=====	=====	=====	=====	=====
UPPER LIMIT						
=====	=====	=====	=====	=====	=====	=====
LOWER LIMIT						
=====	=====	=====	=====	=====	=====	=====
EPA SAMPLE NO.						
=====	=====	=====	=====	=====	=====	=====
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						

IS4 (PHN) = Phenanthrene-d10  
 IS5 (CRY) = Chrysene-d12  
 IS6 (PRY) = Perylene-d12

UPPER LIMIT = + 100%  
 of internal standard area.  
 LOWER LIMIT = - 50%  
 of internal standard area..

# Column used to flag internal standard area values with an asterisk

8D  
PESTICIDE EVALUATION STANDARDS SUMMARY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ GC Column ID: \_\_\_\_\_  
 Dates of Analyses: \_\_\_\_\_ to \_\_\_\_\_

Evaluation Check for Linearity

PESTICIDE	CALIBRATION FACTOR EVAL MIX A	CALIBRATION FACTOR EVAL MIX B	CALIBRATION FACTOR EVAL MIX C	%RSD ( $\leq$ 10.0%)
=====	=====	=====	=====	=====
Aldrin				
Endrin				
4,4'-DDT				
DBC				

(1)

(1) If  $> 10.0\%$  RSD, plot a standard curve and determine the ng for each sample in that set from the curve.

Evaluation Check for 4,4'-DDT/Endrin Breakdown  
(percent breakdown expressed as total degradation)

	DATE ANALYZED	TIME ANALYZED	ENDRIN	4,4'-DDT	COMBINED (2)
=====	=====	=====	=====	=====	=====
INITIAL					
01 EVAL MIX B					
02 EVAL MIX B					
03 EVAL MIX B					
04 EVAL MIX B					
05 EVAL MIX B					
06 EVAL MIX B					
07 EVAL MIX B					
08 EVAL MIX B					
09 EVAL MIX B					
10 EVAL MIX B					
11 EVAL MIX B					
12 EVAL MIX B					
13 EVAL MIX B					
14 EVAL MIX B					

(2) See Form instructions.



8E  
PESTICIDE EVALUATION STANDARDS SUMMARY  
Evaluation of Retention Time Shift for Dibutylchloroendate

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ GC Column ID: \_\_\_\_\_  
 Dates of Analyses: \_\_\_\_\_ to \_\_\_\_\_

	EPA SAMPLE NO.	LAB SAMPLE ID	DATE ANALYZED	TIME ANALYZED	% D	*
01						
02						
03						
04						
05						
06						
07						
08						
09						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						
22						
23						
24						
25						
26						
27						
28						
29						
30						
31						
32						
33						
34						
35						
36						
37						
38						

\* Values outside of QC limits (2.0% for packed columns,  
0.3% for capillary columns)

9  
PESTICIDE/PCB STANDARDS SUMMARY

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Instrument ID: \_\_\_\_\_ GC Column ID: \_\_\_\_\_

DATE(S) OF ANALYSIS FROM: _____ TO: _____ TIME(S) OF ANALYSIS FROM: _____ TO: _____	DATE OF ANALYSIS _____ TIME OF ANALYSIS _____ EPA SAMPLE NO. (STANDARD) _____
--	---

COMPOUND	RT	RT WINDOW		CALIBRATION FACTOR	RT	CALIBRATION FACTOR	QNT Y/N	%D
		FROM	TO					
alpha-BHC								
beta-BHC								
delta-BHC								
gamma-BHC								
Heptachlor								
Aldrin								
Hept. epoxide								
Endosulfan I								
Dieldrin								
4,4'-DDE								
Endrin								
Endosulfan II								
4,4'-DDD								
Endo. sulfate								
4,4'-DDT								
Methoxychlor								
Endrin ketone								
a. Chlordane								
g. Chlordane								
Toxaphene								
Aroclor-1016								
Aroclor-1221								
Aroclor-1232								
Aroclor-1242								
Aroclor-1248								
Aroclor-1254								
Aroclor-1260								

Under QNT Y/N: enter Y if quantitation was performed, N if not performed.  
 %D must be less than or equal to 15.0% for quantitation, and less than or equal to 20.0% for confirmation.

Note: Determining that no compounds were found above the CRQL is a form of quantitation, and therefore at least one column must meet the 15.0% criteria.

For multicomponent analytes, the single largest peak that is characteristic of the component should be used to establish retention time and %D. Identification of such analytes is based primarily on pattern recognition.

10  
PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

GC Column ID (1): \_\_\_\_\_ GC Column ID (2): \_\_\_\_\_

Instrument ID (1): \_\_\_\_\_ Instrument ID (2): \_\_\_\_\_

Lab Sample ID: \_\_\_\_\_

Lab File ID: \_\_\_\_\_ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME	RT WINDOW OF STANDARD From TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 _____	Column 1 _____	_____	-	-
02 _____	Column 2 _____	_____	-	-
03 _____	Column 1 _____	_____	-	-
04 _____	Column 2 _____	_____	-	-
05 _____	Column 1 _____	_____	-	-
06 _____	Column 2 _____	_____	-	-
07 _____	Column 1 _____	_____	-	-
08 _____	Column 2 _____	_____	-	-
09 _____	Column 1 _____	_____	-	-
10 _____	Column 2 _____	_____	-	-
11 _____	Column 1 _____	_____	-	-
12 _____	Column 2 _____	_____	-	-

Comments: \_\_\_\_\_

## **RAS INORGANICS DELIVERY REQUIREMENTS**

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**RAS Inorganics  
Delivery Requirements**

**A. Contract Start-Up Plan**

The contract laboratory must submit a start-up plan for PO approval that details the laboratory's proposed schedule for receiving samples. The laboratory will be required to receive samples within thirty days of contract award.

**B. Updated Standard Operating Procedures**

The contract laboratory must submit updated copies of all required SOPs that were submitted prior to contract award. The updated SOPs must address any and all issues of performance and operation identified during the preaward evaluation process.

**C. Sample Traffic Reports**

The original Sample TR must be returned to SMO with lab receipt information for each sample in the SDG. TRs must be submitted in SDG sets with an SDG coversheet attached.

**D. Sample Data Package**

The sample data package shall include data for analysis of all samples in one SDG, including field samples, reanalyses, blanks, matrix spikes, matrix spike duplicates, and laboratory control samples. The sample data package must include the following:

**1. Cover Page**

The cover page must include: laboratory name; laboratory code; contract number; Case number; SDG number; SOW number; EPA sample numbers in alphanumeric order; detailed documentation of any problems encountered in processing the samples; and completion of the statement on use of ICP background and interelement corrections for the samples.

**2. Sample Data**

**a. Results -- Inorganic Analysis Data Sheet [FORM I - IN]**

Tabulated analytical results (identification and quantitation) of the specified analytes. Appropriate concentration units must be specified and entered on Form I.

**b. Quality Control Data**

(1) Initial and Continuing Calibration Verification [FORM II (PART 1) - IN]

(2) CRDL Standard for AA and Linear Range Analysis for ICP [FORM II (PART 2) - IN]

(3) Blanks [FORM III - IN]

(4) ICP Interference Check Sample [FORM IV - IN]

- (5) Spike Sample Recovery [FORM V (PART 1) - IN]
- (6) Post Digest Spike Sample Recovery [FORM V (PART 2) - IN]
- (7) Duplicates [FORM VI - IN]
- (8) Laboratory Control Sample [FORM VII - IN]
- (9) Standard Addition Results [FORM VIII - IN]
- (10) ICP Serial Dilutions [FORM IX - IN]
- (11) Preparation Log [Form XIII - IN]
- (12) Analysis Run Log [Form XIV - IN]

**c. Quarterly Verification of Instrument Parameters**

- (1) Instrument Detection Limits (Quarterly) [FORM X - IN]
- (2) ICP Interelement Correction Factors (Annually) [FORM XI (PART 1) - IN]
- (3) ICP Interelement Correction Factors (Annually) [FORM XI (PART 2) - IN]
- (4) ICP Linear Ranges (Quarterly) [FORM XII - IN]

(Copies of Quarterly Verification of Instrument Parameters forms for the current quarter must be submitted with each data package.)

**d. Raw Data**

For each reported value, all raw data used to obtain that value must be included in the data package. This applies to all required QA/QC measurements, instrument standardization, as well as all sample analysis results. This statement does not apply to the Quarterly Verification of Instrument Parameters submitted as a part of each data package. Raw data must contain all instrument readouts used for the sample results. Each exposure or instrumental reading must be provided, including those readouts that may fall below the IDL. All AA and ICP instruments must provide a legible hard copy of the direct real-time instrument readout (i.e., stripcharts, printer tapes, etc.). A photocopy of the instruments direct sequential readout must be included. A hardcopy of the instrument's direct instrument readout for cyanide must be included if the instrumentation has the capability.

Raw data in the data package must be ordered as follows: ICP, Flame AA, Furnace AA, Mercury, and Cyanide. All raw data must include concentration units for ICP and absorbances with concentration units for flame AA, furnace AA, Mercury and Cyanide. All flame and furnace AA data must be grouped by element.

Raw data must be labeled with EPA sample number and appropriate codes, shown in Table 1 following, to identify:

- (1) Calibration standards, including source and prep date
- (2) Initial and continuing calibration blanks and preparation blanks
- (3) Initial and continuing calibration verification standards, interference check samples, ICP serial dilution samples, CRDL Standard for ICP and AA, Laboratory Control Sample and Post Digestion Spike
- (4) Diluted and undiluted samples and all weights, dilutions and volumes used to obtain the reported values
- (5) Duplicates
- (6) Spikes
- (7) Instrument used, any instrument adjustments, data corrections or other apparent anomalies on the measurement record, including all data voided or data not used to obtain reported values and a brief written explanation
- (8) All information for furnace analysis clearly and sequentially identified on the raw data, including EPA sample number, sample and analytical spike data, percent recovery, coefficient of variation, full MSA data, MSA correlation coefficient, slope and intercepts of linear fit, final sample concentration (standard addition concentration), and type of background correction used: BS for Smith-Heftje, BD for Deuterium Arc, or BZ for Zeeman
- (9) Time and date of each analysis
- (10) Integration times for AA analyses

e. Digestion and Distillation Logs

Logs shall be submitted in the following order: digestion logs for ICP, flame AA, furnace AA and mercury preparations, followed by a copy of the distillation log for cyanide.

3. A copy of the Sample TRs in Item C must be submitted for all of the samples in an SDG. The TRs must be arranged in increasing EPA sample number order.

E. Data in Computer Readable Form

The contract laboratory must provide a computer-readable copy of the data on data reporting Forms I-XIV for all samples in an SDG. Computer-readable data deliverables shall be submitted on IBM or IBM-compatible, 5.25 inch floppy double-sided, double density 360 K-byte or a high density 1.2 M-byte diskette. The data must be recorded in ASCII, text file format, and must adhere to the file, record and field specifications listed in the SOW.



**Table 1**  
**Codes for Labelling Raw Data**

---

Sample	XXXXXX
Duplicate	XXXXXXD
Matrix Spike	XXXXXXS
Serial Dilution	XXXXXXL
Analytical Spike	XXXXXXA
Post Digestion/Distillation Spike	XXXXXXA
MSA:	
Zero Addition	XXXXXX0
First Addition	XXXXXX1
Second Addition	XXXXXX2
Third Addition	XXXXXX3
Instrument Calibration Standards:	
ICP	S or S0 for blank standard
Atomic Absorption and Cyanide	S0, S10,...etc.
Initial Calibration Verification	ICV
Initial Calibration Blank	ICB
Continuing Calibration Verification	CCV
Continuing Calibration Blank	CCB
Interference Check Samples:	
Solution A	ICSA
Solution AB	ICSAB
CRDL Standard for AA	CRA
CRDL Standard for ICP	CRI
Laboratory Control Samples:	
Aqueous (Water)	LCSW
Solid (Soil/Sediment)	LCSS
Preparation Blank (Water)	PBW
Preparation Blank (Soil)	PBS
Linear Range Analysis Standard	LRS

---

**Notes:**

1. When an analytical spike or MSA is performed on samples other than field samples, the "A", "0", "1", "2" or "3" suffixes must be the last to be added to the EPA Sample Number. For instance, an analytical spike of a duplicate must be formatted "XXXXXXDA."
  2. The numeric suffix that follows the "S" suffix for the standards indicates the true value of the concentration of the standard in ug/L.
  3. ICP calibration standards usually consist of several analytes at different concentrations. Therefore, no numeric suffix can follow the ICP calibration standards unless all the analytes in the standard are prepared at the same concentrations. For instance, the blank for ICP must be formatted "S0."
  4. The CRDL standard for AA is considered to be a calibration standard if it was a part of the calibration curve, thus it must be formatted like any other standard. The "CRA" format must be used if the CRDL standard for AA is not used to establish the calibration curve.
-

F. Results of Intercomparison/Performance Evaluation Sample Analyses

Tabulation of analytical results for Intercomparison/PE Sample analyses include all requirements specified in Items D and E.

G. Complete Case File Purge

The complete case file purge includes all laboratory records received or generated for a specific Case that have not been previously submitted to EPA as a deliverable. These items include but are not limited to: sample tags, custody records, sample tracking records, analysts logbook pages, bench sheets, instrument readout records, computer printouts, raw data summaries, instrument logbook pages (including instrument conditions), correspondence, and the document inventory.

I. Quarterly Verification of Instrument Parameters

The contract laboratory must perform and report quarterly verification of instrument detection limits and linear range by methods specified in the SOW for each instrument used. For the ICP instrumentation and methods, the contract laboratory must also report quarterly interelement correction factors (including method of determination), wavelengths used and integration times. Quarterly Verification of Instrument Parameters forms for the current quarter must be submitted in each SDG data package, using Forms X, XI and XII. Submission of Quarterly Verification of Instrument Parameters must include the raw data used to determine those values reported.

**RAS INORGANICS  
DATA REPORTING FORMS**

## COVER PAGE - INORGANIC ANALYSES DATA PACKAGE

Lab Sample ID.

[illegible]

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## U.S. EPA - CLP

1  
INORGANIC ANALYSIS DATA SHEET

EPA SAMPLE NO.

--

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix (soil/water): \_\_\_\_\_ Lab Sample ID: \_\_\_\_\_

Level (low/med): \_\_\_\_\_ Date Received: \_\_\_\_\_

% Solids: \_\_\_\_\_

Concentration Units (ug/L or mg/kg dry weight): \_\_\_\_\_

CAS No.	Analyte	Concentration	C	Q	M
7429-90-5	Aluminum				
7440-36-0	Antimony				
7440-38-2	Arsenic				
7440-39-3	Barium				
7440-41-7	Beryllium				
7440-43-9	Cadmium				
7440-70-2	Calcium				
7440-47-3	Chromium				
7440-48-4	Cobalt				
7440-50-8	Copper				
7439-89-6	Iron				
7439-92-1	Lead				
7439-95-4	Magnesium				
7439-96-5	Manganese				
7439-97-6	Mercury				
7440-02-0	Nickel				
7440-09-7	Potassium				
7782-49-2	Selenium				
7440-22-4	Silver				
7440-23-5	Sodium				
7440-28-0	Thallium				
7440-62-2	Vanadium				
7440-66-6	Zinc				
	Cyanide				

Color Before: \_\_\_\_\_ Clarity Before: \_\_\_\_\_ Texture: \_\_\_\_\_

Color After: \_\_\_\_\_ Clarity After: \_\_\_\_\_ Artifacts: \_\_\_\_\_

Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

## U.S. EPA - CLP

2A

## INITIAL AND CONTINUING CALIBRATION VERIFICATION

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Initial Calibration Source: \_\_\_\_\_

Continuing Calibration Source: \_\_\_\_\_

Concentration Units: ug/L

Analyte	Initial Calibration			Continuing Calibration					M
	True	Found	%R(1)	True	Found	%R(1)	Found	%R(1)	
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

(1) Control Limits: Mercury 80-120; Other Metals 90-110; Cyanide 85-115

## U.S. EPA - CLP

2B

## CRDL STANDARD FOR AA AND ICP

Lab Name: \_\_\_\_\_

Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_

Case No.: \_\_\_\_\_

SAS No.: \_\_\_\_\_

SDG No.: \_\_\_\_\_

AA CRDL Standard Source: \_\_\_\_\_

ICP CRDL Standard Source: \_\_\_\_\_

Concentration Units: ug/L

Analyte	CRDL Standard for AA			CRDL Standard for ICP				
	True	Found	%R	True	Initial Found	%R	Final Found	%R
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								

## U.S. EPA - CLP

3  
BLANKS

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Preparation Blank Matrix (soil/water): \_\_\_\_\_

Preparation Blank Concentration Units (ug/L or mg/kg): \_\_\_\_\_

Analyte	Initial Calib. Blank (ug/L)	C	Continuing Calibration Blank (ug/L)						Prepa- ration Blank	C	M
			1	C	2	C	3	C			
Aluminum											
Antimony											
Arsenic											
Barium											
Beryllium											
Cadmium											
Calcium											
Chromium											
Cobalt											
Copper											
Iron											
Lead											
Magnesium											
Manganese											
Mercury											
Nickel											
Potassium											
Selenium											
Silver											
Sodium											
Thallium											
Vanadium											
Zinc											
Cyanide											



## U.S. EPA - CLP

4

## ICP INTERFERENCE CHECK SAMPLE

Lab Name: \_\_\_\_\_

Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_

Case No: \_\_\_\_\_

SAS No.: \_\_\_\_\_

SDG No.: \_\_\_\_\_

ICP ID Number: \_\_\_\_\_

ICS Source: \_\_\_\_\_

Concentration Units: ug/L

Analyte	True		Initial Found			Final Found		
	Sol. A	Sol. AB	Sol. A	Sol. AB	%R	Sol. A	Sol. AB	%R
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								

## U.S. EPA - CLP

5A  
SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix (soil/water): \_\_\_\_\_ Level (low/med): \_\_\_\_\_

Concentration Units (ug/L or mg/kg dry weight): \_\_\_\_\_

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

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U.S. EPA - CLP

5B  
POST DIGEST SPIKE SAMPLE RECOVERY

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_  
 Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_  
 Matrix (soil/water): \_\_\_\_\_ Level (low/med): \_\_\_\_\_

Concentration Units: ug/L

Analyte	Control Limit %R	Spiked Sample Result (SSR)	C	Sample Result (SR)	C	Spike Added (SA)	%R	Q	M
Aluminum									
Antimony									
Arsenic									
Barium									
Beryllium									
Cadmium									
Calcium									
Chromium									
Cobalt									
Copper									
Iron									
Lead									
Magnesium									
Manganese									
Mercury									
Nickel									
Potassium									
Selenium									
Silver									
Sodium									
Thallium									
Vanadium									
Zinc									
Cyanide									

Comments:

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## U.S. EPA - CLP

6  
DUPLICATES

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix (soil/water): \_\_\_\_\_ Level (low/med): \_\_\_\_\_

% Solids for Sample: \_\_\_\_\_ % Solids for Duplicate: \_\_\_\_\_

Concentration Units (ug/L or mg/kg dry weight): \_\_\_\_\_

Analyte	Control Limit	Sample (S)	C	Duplicate (D)	C	RPD	Q	M
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								

## U.S. EPA - CLP

7

## LABORATORY CONTROL SAMPLE

Lab Name: \_\_\_\_\_

Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_

Case No.: \_\_\_\_\_

SAS No.: \_\_\_\_\_

SDG No.: \_\_\_\_\_

Solid LCS Source: \_\_\_\_\_

Aqueous LCS Source: \_\_\_\_\_

Analyte	Aqueous (ug/L)			Solid (mg/kg)				
	True	Found	%R	True	Found	C	Limits	%R
Aluminum								
Antimony								
Arsenic								
Barium								
Beryllium								
Cadmium								
Calcium								
Chromium								
Cobalt								
Copper								
Iron								
Lead								
Magnesium								
Manganese								
Mercury								
Nickel								
Potassium								
Selenium								
Silver								
Sodium								
Thallium								
Vanadium								
Zinc								
Cyanide								



## U.S. EPA - CLP

9  
ICP SERIAL DILUTIONS

EPA SAMPLE NO.

Lab Name: \_\_\_\_\_ Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_ SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

Matrix (soil/water): \_\_\_\_\_ Level (low/med): \_\_\_\_\_

Concentration Units: ug/L

Analyte	Initial Sample Result (I)	C	Serial Dilution Result (S)	C	% Differ- ence	Q	M
Aluminum							
Antimony							
Arsenic							
Barium							
Beryllium							
Cadmium							
Calcium							
Chromium							
Cobalt							
Copper							
Iron							
Lead							
Magnesium							
Manganese							
Mercury							
Nickel							
Potassium							
Selenium							
Silver							
Sodium							
Thallium							
Vanadium							
Zinc							

10  
HOLDING TIMES

Lab Code: Case No.: SAS No.: SDG No.:

[illegible]



## U.S. EPA - CLP

11  
INSTRUMENT DETECTION LIMITS (QUARTERLY)

Lab Name: \_\_\_\_\_

Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_

Case No.: \_\_\_\_\_

SAS No.: \_\_\_\_\_

SDG No.: \_\_\_\_\_

ICP ID Number: \_\_\_\_\_

Date: \_\_\_\_\_

Flame AA ID Number: \_\_\_\_\_

Furnace AA ID Number: \_\_\_\_\_

Analyte	Wave-length (nm)	Back-ground	CRDL (ug/L)	IDL (ug/L)	M
Aluminum			200		
Antimony			60		
Arsenic			10		
Barium			200		
Beryllium			5		
Cadmium			5		
Calcium			5000		
Chromium			10		
Cobalt			50		
Copper			25		
Iron			100		
Lead			5		
Magnesium			5000		
Manganese			15		
Mercury			0.2		
Nickel			40		
Potassium			5000		
Selenium			5		
Silver			10		
Sodium			5000		
Thallium			10		
Vanadium			50		
Zinc			20		

Comments:

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## U.S. EPA - CLP

12A

## ICP INTERELEMENT CORRECTION FACTORS (QUARTERLY)

Lab Name: \_\_\_\_\_

Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_

Case No.: \_\_\_\_\_

SAS No.: \_\_\_\_\_

SDG No.: \_\_\_\_\_

ICP ID Number: \_\_\_\_\_

Date: \_\_\_\_\_

Analyte	Wave-length (nm)	Interelement Correction Factors for:				
		Al	Ca	Fe	Mg	___
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

Comments:

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## U.S. EPA - CLP

12B

## ICP INTERELEMENT CORRECTION FACTORS (QUARTERLY)

Lab Name: \_\_\_\_\_

Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_

Case No.: \_\_\_\_\_

SAS No.: \_\_\_\_\_

SDG No.: \_\_\_\_\_

ICP ID Number: \_\_\_\_\_

Date: \_\_\_\_\_

Analyte	Wave-length (nm)	Interelement Correction Factors for:				
		—	—	—	—	—
Aluminum						
Antimony						
Arsenic						
Barium						
Beryllium						
Cadmium						
Calcium						
Chromium						
Cobalt						
Copper						
Iron						
Lead						
Magnesium						
Manganese						
Mercury						
Nickel						
Potassium						
Selenium						
Silver						
Sodium						
Thallium						
Vanadium						
Zinc						

Comments:

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## U.S. EPA - CLP

13

## ICP LINEAR RANGES (QUARTERLY)

Lab Name: \_\_\_\_\_

Contract: \_\_\_\_\_

Lab Code: \_\_\_\_\_ Case No.: \_\_\_\_\_

SAS No.: \_\_\_\_\_ SDG No.: \_\_\_\_\_

ICP ID Number: \_\_\_\_\_

Date: \_\_\_\_\_

Analyte	Integ. Time (Sec.)	Concentration (ug/L)	M
Aluminum			
Antimony			
Arsenic			
Barium			
Beryllium			
Cadmium			
Calcium			
Chromium			
Cobalt			
Copper			
Iron			
Lead			
Magnesium			
Manganese			
Mercury			
Nickel			
Potassium			
Selenium			
Silver			
Sodium			
Thallium			
Vanadium			
Zinc			

Comments:

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## **RAS DIOXIN DELIVERY REQUIREMENTS**

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**RAS Dioxin  
Delivery Requirements**

**A. Dioxin Shipment Record**

The contract laboratory must submit the original Sample DSR with lab receipt information.

**B. Sample Data Summary Package**

The contract laboratory is required to submit a hard copy of analytical data and documentation from the sample data package as follows:

1. Case Narrative
2. Completed data reporting sheets consisting of Forms B-1, B-2, B-3 and B-4. Original and rerun sample data must be provided on Form B-1.

**C. Sample Data Package**

Hard copy analytical data and documentation are required as described below:

1. The Case Narrative must contain: the Case number, DSR numbers, contract number and detailed documentation of any quality control, sample shipment and/or analytical problems encountered in a specific Case.
2. Copies of completed DSRs for all samples reported in the data package.
3. Results of initial triplicate analyses of four concentration calibration solutions, including all Selected Ion Current Profiles, Calculated Response Factors, plotted concentration calibration curves and computer generated quantitation reports.
4. Completed data reporting sheets (Forms B-1, B-2, B-3 and B-4) with appropriate SICPs.
5. SICPs generated during each performance check solution analysis and each concentration calibration solution analysis.
6. A chronological list of all analyses performed. If more than one GC/MS system is used, a chronological list is required for each system.

**D. Monthly Sample Status Report**

The contract laboratory is required to provide the status of all samples received or in-house during the calendar month. Required status information includes: samples received, samples extracted, samples analyzed, and samples rerun. All samples must be identified by appropriate EPA Sample, Case and Batch/Shipment numbers.

**E. Daily Sample Status Report**

In response to verbal request from SMO or the PO, the contract laboratory must verbally provide sample status information on a same-day basis. Should written confirmation be requested, the laboratory must send daily sample status information in written form that same day using first-class mail service.

**F. GC/MS Tapes**

The contract laboratory must store all raw GC/MS data on magnetic tape, in appropriate manufacturer's format. This tape must include: samples, blanks, concentration calibration solutions, and performance evaluation samples. The laboratory must maintain a written reference/logbook of tape files to EPA sample number, calibration data, standards and blanks.

**G. Extracts and Unused Sample Volume**

The contract laboratory must retain extracts, stored at 4°C, for 365 days after data submission. Unused sample volume must also be retained, stored at ambient temperature, for 365 days after data submission.

**H. Complete Case File Purge**

The complete case file purge includes all laboratory records received or generated for a sample batch that have not been previously submitted to EPA as a deliverable. These items include but are not limited to: sample tags, custody records, sample tracking records, analysts logbook pages, bench sheets, chromatographic charts, computer printouts, raw data summaries, instrument logbook pages, correspondence, and the document inventory.



**RAS DIOXIN  
DATA REPORTING FORMS**









A. TCDD REPORT FORM (Form B-1)

This form is used for tabulating and reporting case results.

Complete the header information at the top of the page including instrument ID, laboratory name, case/batch number, report date, and column used.

EPA sample number is tabulated along with date sample was extracted, and weight (wet) extracted to the nearest tenth (0.1) of a gram or volume extracted (water) to the nearest 10 milliliters.

Calculate the concentration of 2,3,7,8-TCDD using the formula:

$$C_x = \frac{A_x \cdot Q_{IS}}{A_{IS} \cdot RRF_n \cdot W}$$

$C_x$  = 2,3,7,8-TCDD concentration in ug/kg or ug/L

$A_x$  = the sum of integrated ion abundance detected for m/z 320 and 322

$A_{IS}$  = the sum of integrated ion abundances detected for m/z 332 and 334 (characteristic ions of  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD the internal standard).

$Q_{IS}$  = quantity (in ng) of  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD added to the sample before extraction

$RRF_n$  = calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD

$W$  = The weight (in g) of soil/sediment extracted or volume of water extracted (in mL)

Positive samples are quantitated with values >10.0 ug/kg or 100 ng/L recorded to three (3) significant figures and those values <10.0 ug/kg or 100 ng/L reported to two (2) significant figures.

For samples in which unlabeled 2,3,7,8-TCDD was not detected calculate the estimated maximum possible concentration, which is the concentration required to produce a signal with a peak height of 2.5 times the background signal height.

Use the formula:

$$MPC = \frac{2.5 \cdot H_x \cdot Q_{IS}}{H_{IS} \cdot RRF_n \cdot W}$$

where: MPC = maximum possible concentration of unlabeled 2,3,7,8-TCDD required to produce  $H_x$ .

$H_x$  = peak height for m/z 320 or 322 in the same group of >5 scans used to measure  $A_{is}$ .

$H_{is}$  = peak height for the appropriate ion characteristic of the internal standard, m/z 332 when 320 is used to determine  $A_x$ , and m/z 334 when 322 is used to determine  $A_x$ .

$Q_{is}$  = quantity (in ng) of  $^{13}C_{12}$ -2,3,7,8-TCDD added to the sample before extraction.

$RRF_n$  = calculated mean response factor for unlabeled 2,3,7,8-TCDD relative to  $^{13}C_{12}$ -2,3,7,8-TCDD.

$W$  = weight (in g) of wet soil/sediment sample or volume of water extracted (in mL).

Report GC/MS Instrument ID, the date and time the analysis was performed, and the signal to noise ratio for the surrogate compound.

## INITIAL CALIBRATION SUMMARY

Laboratory: \_\_\_\_\_ CC Solution Alternative: \_\_\_\_\_

Case/Batch No.: \_\_\_\_\_ Instrument ID: \_\_\_\_\_

Date	Time	Sol. ID	AREA						
			320	322	328	332IS	334IS	332RS	334RS
		CC1 CC1 CC1							
		CC2 CC2 CC2							
		CC3 CC3 CC3			*				
		CC4 CC4 CC4			↑ ↑ / / ↑ ↑				

Solution ID Codes:

CC1 = Concentration calibration solution #1  
 CC2 = Concentration calibration solution #2  
 CC3 = Concentration calibration solution #3  
 CC4 = Concentration calibration solution #4

\* Not present in CC Solution  
 Alternative One.



## INITIAL CALIBRATION SUMMARY

Laboratory: \_\_\_\_\_ CC Solution Alternative: \_\_\_\_\_

Case/Batch No.: \_\_\_\_\_ Instrument ID: \_\_\_\_\_

Date	Time	Sol. ID	Measured RRF <sub>n</sub>	Mean RRF <sub>n</sub>	Measured RRF <sub>i</sub>	Mean RRF <sub>i</sub>
		CC1 CC1 CC1				
		CC2 CC2 CC2				
		CC3 CC3 CC3				
		CC4 CC4 CC4				

Solution ID Codes:

CC1 = Concentration calibration solution #1

CC2 = Concentration calibration solution #2

CC3 = Concentration calibration solution #3

CC4 = Concentration calibration solution #4

ZRSD: RRF<sub>n</sub>      RRF<sub>i</sub>

CC1= \_\_\_\_\_

CC2= \_\_\_\_\_

CC3= \_\_\_\_\_

CC4= \_\_\_\_\_

Native Mean  
of Means: \_\_\_\_\_

IS Mean  
of Means: \_\_\_\_\_

B. Initial Calibration Summary (Form B-2)

Record all routine calibrations (PCS and CCI) performed during initial calibration on form B-3.

Complete all header information including laboratory, case/batch number, and instrument ID and EPA CC Solution Alternative.

Date and time along with response for each ion is recorded for each calibration solution. The response factors are calculated with the following equations:

$RRF_n$  (native Response Factor)

$RRF_i$  (internal Standard Response Factor)

$$RRF_n = \frac{A_x \cdot Q_{is}}{A_{is} \cdot Q_n}$$

$$RRF_i = \frac{A_{is} \cdot Q_{rs}}{A_{rs} \cdot Q_{is}}$$

Where:

$A_x$  = the sum of integrated ion abundance of m/z 320 and 322 for unlabeled 2,3,7,8-TCDD

$A_{is}$  = the sum of integrated ion abundances of m/z 332 and m/z 334 for  $^{13}C_{12}$ -2,3,7,8-TCDD

$A_{rs}$  = the sum of integrated ion abundance of m/z 332 and m/z 334 for  $^{13}C_{12}$ -1,2,3,4-TCDD

$Q_n$  = quantity of unlabeled 2,3,7,8-TCDD injected

$Q_{is}$  = quantity of  $^{13}C_{12}$ -2,3,7,8-TCDD injected

$Q_{rs}$  = quantity of  $^{13}C_{12}$ -1,2,3,4-TCDD

Calculate the mean RRF and the percent relative standard deviation for the triplicate runs of each calibration solution.

$$\%RSD = \frac{SD}{\bar{X}} \times 100$$

Where:

$$SD = \sqrt{\frac{\sum_{i=1}^N (X_i - \bar{X})^2}{N - 1}}$$

$\bar{X}$  = mean of each of the three Response Factors respectively

From the 4 mean native response factors and 4 mean internal standard response factors: calculate the mean of means for each respective RRF's.

## FORM B-3

## ROUTINE CALIBRATION SUMMARY

Laboratory: \_\_\_\_\_

CC Solution Alternative: \_\_\_\_\_

Case/Batch No.: \_\_\_\_\_

Instrument ID: \_\_\_\_\_

(PCS) PERFORMANCE CHECK SOL. (CCI)  
CON. CALIB. SOL. #1

Date									
Time									
Response									
259									
320									
322									
328									
332IS									
334IS									
332RS									
334RS									
Ratios									
320/322									
332/334IS									
332/334RS									
RRF <sub>n</sub>	—	—	—	—	—	—			
RRF <sub>i</sub>	—	—	—	—	—	—			
% Valley							—	—	—

C. Routine Calibration Summary (Form B-3)

Complete the header information including the laboratory, instrument ID Case/Batch number and EPA CC Solution Alternative.

For each performance check solution analyzed complete the date and time of analysis, the response for m/z 259, 320, and 322 for unlabeled 2,3,7,8-TCDD, 328 for  $^{37}\text{Cl}_4$ -2,3,7,8-TCDD, and 332 and 334 for  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD and  $^{13}\text{C}_{12}$ -1,2,3,4-TCDD.

Ion ratios for m/z 320/322, m/z 332/334 for  $^{13}\text{C}_{12}$ -2,3,7,8-TCDD and m/z 332/334 for  $^{13}\text{C}_{12}$ -1,2,3,4-TCDD are to be calculated and recorded.

Response factors are to be calculated as in the Initial Calibration Summary (Section B).

For calculation of valley percent see Section D, Section 9.2.6.1.

For each Concentration Calibration Solution #1 used in Routine Calibration, complete all the above information.

QUALITY CONTROL SUMMARY

Laboratory Name \_\_\_\_\_

Case/Batch No. \_\_\_\_\_

Instrument ID \_\_\_\_\_

SOIL

Accuracy, Fortified/  
Spike Field Blank: \_\_\_\_\_

EPA Sample Number: \_\_\_\_\_

Relative Difference (%),  
Duplicate Analysis: \_\_\_\_\_

EPA Sample Number: \_\_\_\_\_

WATER

Accuracy, Fortified/  
Spike Field Blank: \_\_\_\_\_

EPA Sample Number: \_\_\_\_\_

Relative Difference (%),  
Duplicate Analysis: \_\_\_\_\_

EPA Sample Number: \_\_\_\_\_

D. QC Summary

Complete all the header information.

Report the sample number for the fortified field blank and the % accuracy of the fortified/spike field blank by using the following equation:

$$\% \text{ accuracy} = \frac{\text{amount measured}}{1.0} \times 100$$

Record the sample used for duplicate and the Relative Percent Difference which is calculated as follows:

$$\text{RPD} = \frac{\frac{|S_1 - S_2|}{S_1 - S_2}}{2} \times 100$$

Where:

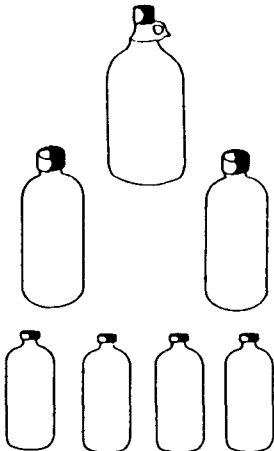


$S_1$  and  $S_2$  represent sample and duplicate sample results.



## **APPENDIX D**

### **SAMPLE INFORMATION AND DOCUMENTATION**



# ORGANIC SAMPLE COLLECTION REQUIREMENTS

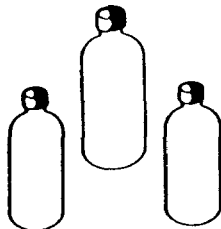

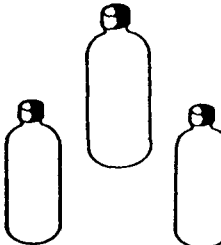

WATER SAMPLES	REQUIRED VOLUME		CONTAINER TYPE
EXTRACTABLE ANALYSIS (LOW LEVEL)	1 GALLON		1 x 4-LITER AMBER GLASS BOTTLES OR 2 x 80-OZ. AMBER GLASS BOTTLES OR 4 x 1-LITER AMBER GLASS BOTTLES
EXTRACTABLE ANALYSIS (MEDIUM LEVEL*)	1 GALLON		4 x 32-OZ. WIDE-MOUTH GLASS JARS
VOLATILE ANALYSIS (LOW OR MEDIUM LEVEL*)	80 ML		2 x 40-ML GLASS VIALS


SOIL/SEDIMENT SAMPLES	REQUIRED VOLUME		CONTAINER TYPE
EXTRACTABLE ANALYSIS (LOW OR MEDIUM LEVEL*)	6 OZ.		1 x 8-OZ. WIDE-MOUTH GLASS JAR OR 2 x 4-OZ. WIDE-MOUTH GLASS JARS
VOLATILE ANALYSIS (LOW OR MEDIUM LEVEL*)	240 ML		2 x 120-ML WIDE-MOUTH GLASS VIALS

\*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT

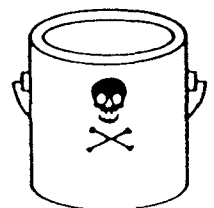


# INORGANIC SAMPLE COLLECTION REQUIREMENTS

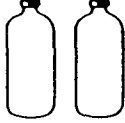
WATER SAMPLES	REQUIRED VOLUME		CONTAINER TYPE
METALS ANALYSIS (LOW LEVEL)	1 LITER		1 x 1-LITER POLYETHYLENE BOTTLE OR 2 x 500 ML POLYETHYLENE BOTTLE
METALS ANALYSIS (MEDIUM LEVEL*)	16 OZ.		1 x 16-OZ. WIDE-MOUTH GLASS JAR
CYANIDE (CN <sup>-</sup> ) ANALYSIS (LOW LEVEL)	1 LITER		1 x 1-LITER POLYETHYLENE BOTTLE OR 2 x 500 ML POLYETHYLENE BOTTLE
CYANIDE (CN <sup>-</sup> ) ANALYSIS (MEDIUM LEVEL*)	16 OZ.		1 x 16-OZ. WIDE-MOUTH GLASS JAR



SOIL/SEDIMENT SAMPLES	REQUIRED VOLUME		CONTAINER TYPE
METALS AND CYANIDE (CN <sup>-</sup> ) ANALYSIS (LOW OR MEDIUM LEVEL*)	6 OZ.		1 x 8-OZ. WIDE-MOUTH GLASS JAR OR 2 x 4-OZ. WIDE-MOUTH GLASS JARS

\*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT




# DIOXIN SAMPLE COLLECTION REQUIREMENTS

WATER SAMPLES	REQUIRED VOLUME		CONTAINER TYPE
2,3,7,8-TCDD ANALYSIS (MULTI-CONCENTRATION)	2 LITERS		2 × 1-LITER AMBER GLASS BOTTLES

SOIL/SEDIMENT SAMPLES	REQUIRED VOLUME		CONTAINER TYPE
2,3,7,8-TCDD ANALYSIS (MULTI-CONCENTRATION)	4 OZ.		1 × 4-OZ. WIDE-MOUTH GLASS JAR
			OR 1 × 8-OZ. WIDE-MOUTH GLASS JAR

# HIGH HAZARD SAMPLE COLLECTION REQUIREMENTS

LIQUID OR SOLID SAMPLES	REQUIRED VOLUME		CONTAINER TYPE
ORGANIC AND INORGANIC ANALYSIS	6 OZ.		1 × 8-OZ. WIDE-MOUTH GLASS JAR

\*ALL MEDIUM LEVEL SAMPLES TO BE SEALED IN METAL PAINT CAN FOR SHIPMENT



U.S. ENVIRONMENTAL PROTECTION AGENCY  
CLP Sample Management Office  
P.O. Box 818 - Alexandria, Virginia 22313  
Phone: 703/557-2490 - FTS/557-2490

SAS Number

**SPECIAL ANALYTICAL SERVICES**

**Client Request**

☐

Regional Transmittal

☐

Telephone Request

- A. EPA Region/Client: \_\_\_\_\_
- B. RSCC Representative: \_\_\_\_\_
- C. Telephone Number: (    ) \_\_\_\_\_
- D. Date of Request: \_\_\_\_\_
- E. Site Name: \_\_\_\_\_

Please provide below description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in a delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_
2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium or high concentration):  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_
3. Purpose of analysis (specify whether Superfund (enforcement or remedial action), RCRA, NPDES, etc.): \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

4. Estimated date(s) of collection: \_\_\_\_\_  
\_\_\_\_\_
5. Estimated date(s) and method of shipment: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_
6. Number of days analysis and data required after laboratory receipt of samples:  
\_\_\_\_\_
7. Analytical protocol required (attach copy if other than a protocol currently used in this program): \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_
8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.): \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_
9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.) If not completed, format of results will be left to program discretion. \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_
10. Other (use additional sheets or attach supplementary information, as needed):  
\_\_\_\_\_
11. Name of sampling/shipping contact: \_\_\_\_\_  
Phone: (    ) \_\_\_\_\_

12. Data Requirements

<u>Parameter</u>	<u>Detection Limit</u>	<u>Precision Desired (+/-% or Concentration)</u>

13. QC Requirements

<u>Audits Required</u>	<u>Frequency of Audits</u>	<u>Limits (Percent or Concentration)</u>

14. Action Required if Limits are Exceeded


Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

## **SAMPLE DOCUMENTATION**





United States Environmental Protection Agency Contract Laboratory Program Sample Management Office PO Box 818 Alexandria, VA 22313 703-557-2490 FTS 557-2490										Organic Traffic Report (For CLP Use Only)		Case Number 10001	SAS No. (If applicable)
1. Type of Activity (Check one)		2. Region Number		3. Ship To:		4. Date Shipped		5. Sample Description (Enter in Column A)					
<input type="checkbox"/> ENF <input type="checkbox"/> NPLD <input type="checkbox"/> RA <input type="checkbox"/> SI <input type="checkbox"/> STSI <input type="checkbox"/> ER <input type="checkbox"/> O&M <input type="checkbox"/> RD <input type="checkbox"/> ST <input type="checkbox"/> Other (Specify) <input type="checkbox"/> ESI <input type="checkbox"/> PA <input checked="" type="checkbox"/> RIFS <input type="checkbox"/> STPA		I		XYZ Co.		11-4-88		1234567890		1. Surface Water 2. Ground Water 3. Leachate 4. Rinsate 5. Soil/Sediment 6. Oil (SAS) 7. Waste (SAS) 8. Other (SAS) (Specify)			
Non-Superfund Program		Joe Sampler		Carrier		Fed Ex							
Site Name		CLP Lab		Triple volume required for matrix spike/duplicate aqueous sample.									
Drum Site		100 Main St.		Ship medium and high concentration samples in paint cans.									
City, State		Anytown, MA		Attn M.Spec									
Site Spill ID		03											
CLP Sample Number (From labels)	(A) Sample Description (From box 1)	(B) Concentration L=low M=med H=high	(C) RAS Analysis			(D) Special Handling	(E) Station Location	(F) Date/Time of Sample Collection	(G) Corresponding CLP Inorganic Sample Number				
			VOA	BNA	Pest/PCB								
AB123	2	L	X	X	X		MW-1	11-3/0800	MAZ 221				
AB124	2	L	X	X	X		MW-2	11-3/0830	MAZ 222				
AB125	2	L	X	X	X		MW-3	11-3/0845	MAZ 223				
AB126	2	L	X	X	X		MW-4	11-3/0900	MAZ 224				
AB127	2	L	X	X	X		MW-5	11-3/1000	MAZ 225				
AB128	2	L	X	X	X		MW-6	11-3/1030	MAZ 226				
AB129	2	L	X	X	X		MW-7	11-3/1100	MAZ 227				
AB130	2	L	X	X	X		MW-8	11-3/1130	MAZ 228				
AB131	2	L	X	X	X		MW-9	11-3/1400	MAZ 229				
AB132	2	L	X	X	X		MW-10	11-3/1500	MAZ 230				
AB133	2	L	X	X	X		MW-11	11-3/1530	MAZ 231				
AB134	2	L	X	X	X		MW-12	11-3/1600	MAZ 232				
AB135	2	L	X	X	X	MS/MSD	MW-13	11-3/1700	MAZ 233				
AB136	2	L	X	X	X		MW-14	11-4/0900	MAZ 234				
AB137	2	L	X	X	X		MW-15	11-4/1000	MAZ 235				
AB138	2	L	X	X	X		MW-16	11-4/1100	MAZ 236				
—			SHIPPING			COMPLETE							
			No more to ship			under this case no.							



United States Environmental Protection Agency Contract Laboratory Program Sample Management Office PO Box 818 Alexandria, VA 22313 703-557-2490 FTS 557-2490										Inorganic Traffic Report (For CLP Use Only)		Case Number <b>10101</b>	SAS No. (if applicable)
<b>EPA</b> 1. Type of Activity (Check one) <input type="checkbox"/> ENF <input type="checkbox"/> NPLD <input type="checkbox"/> RA <input checked="" type="checkbox"/> SI <input type="checkbox"/> STSI <input type="checkbox"/> ER <input type="checkbox"/> O&M <input type="checkbox"/> RD <input type="checkbox"/> ST <input type="checkbox"/> STPA <input type="checkbox"/> ESI <input type="checkbox"/> PA <input type="checkbox"/> RIFS <input type="checkbox"/> STPA Non-Superfund Program				2. Region Number <input checked="" type="checkbox"/> <b>Acme Co.</b> Sampler (Name) <b>Joan Sampler</b>		4. Date Shipped <b>11-4-88</b> Airbill Number <b>0987654321</b> Carrier <b>Fed Ex</b>		5. Sample Description (Enter in Column A) 1. Surface Water 2. Ground Water 3. Leachate 4. Rinsate 5. Soil/Sediment 6. Oil (SAS) 7. Waste (SAS) 8. Other (SAS) (Specify)					
Site Name <b>Drum Site</b> City/State <b>Green City, OR</b>				3. Ship To: <b>Analytical Lab</b> <b>100 Center Ave</b> <b>Anytown, CA 94568</b> <b>Attn: A. Metal</b>		Double volume required for matrix spike/duplicate aqueous sample. Ship medium and high concentration samples in paint cans. See reverse for additional instructions.							
CLP Sample Number (From label)	(A) Sample Description (From box 1)	(B) Concentration L=low M=med H=high	(C) RAS Analysis		(D) Special Handling	(E) Station Location	(F) Date/Time of Sample Collection	(G) Corresponding Organic Sample Number					
			Total Metals	Cyanide									
MJZ 900	/	L	X	X		LOC-1	11-4/0700	JA 321					
MJZ 901	/	L	X	X		LOC-2	11-4/0730	JA 322					
MJZ 902	/	L	X	X		LOC-3	11-4/0800	JA 323					
MJZ 903	/	L	X	X		LOC-4	11-4/0830	JA 324					
MJZ 904	/	L	X	X		LOC-5	11-4/0900	JA 325					
MJZ 905	/	L	X	X		LOC-6	11-4/0930	JA 326					
MJZ 906	/	L	X	X		LOC-7	11-4/0945	JA 327					
MJZ 907	/	L	X	X		LOC-8	11-4/1000	JA 328					
MJZ 908	/	L	X	X		LOC-9	11-4/1030	JA 329					
MJZ 909	/	L	X	X		LOC-10	11-4/1100	JA 330					
MJZ 910	/	L	X	X		LOC-11	11-4/1130	JA 331					
MJZ 911	/	L	X	X		LOC-12	11-4/1200	JA 332					
MJZ 912	/	L	X	X		LOC-13	11-4/1215	JA 333					
MJZ 913	/	L	X	X		LOC-14	11-4/1245	JA 334					
MJZ 914	/	L	X	X		LOC-15	11-4/1300	JA 335					
MJZ 915	/	L	X	X		LOC-16	11-4/1330	JA 336					
MJZ 916	/	L	X	X		LOC-17	11-4/1400	JA 337					
MJZ 917	/	L	X	X		LOC-18	11-4/1430	JA 338					
MJZ 918	/	L	X	X		LOC-19	11-4/1500	JA 339					
MJZ 920	/	L	X	X	MS/dup	LOC-20	11-4/1530	JA 340					



USEPA Contract Laboratory Program  
Sample Management Office  
P.O. Box 818 Alexandria, Virginia 22313  
FTS 8-557-2490 703/557-2490

CASE NO: 3000

BATCH NO: 03

SAS NO:  
(if applicable) N/A

CLP DIOXIN SHIPMENT RECORD

Type of Activity (circle one) <b>D</b> Superfund — PA SI ESI RD RA ER NPLD O&M OTHER Non-Superfund — Program		Region Number: <b>IV</b>	Ship To: <b>Dioxin Lab</b> <b>100 Oak Run</b> <b>Testtown, OK</b> ATTN: <b>N. Analyst</b> <b>67891</b>
Site Name: <b>Drum Site</b> City, State: <b>Prustville, FL #24</b>	Site Spill ID: <b>#24</b>	Sampling Contact: <b>John Digger</b> (name) <b>Sampling, Inc.</b> (company)	Date Shipped: <b>11/7/88</b>
Sampling Date: <b>11/6/88</b>	Carrier: <b>Fed. Ex.</b> Airbill No: <b>123456789</b>	Sampler Instructions: 1) Ship all samples in paint cans, with sample labels affixed to outside of can. 2) Use TCE or hexane organic solvents for rinsate samples. 3) Sample Volumes Required: <u>Soil or Sediment</u> : 4 oz. per sample in glass jar. <u>Aqueous</u> : 2 Liters per sample in amber glass. Send one 4 Liter sample per Batch of aqueous samples for lab OC. 4) "Sample to spike" will be analyzed at Lab as a spiked sample only. If this sample requires analysis prior to spiking, the sampler must supply a separate sample labelled with a unique sample number.	

CLP SAMPLE NUMBERS (from labels)	MATRIX (check one/sample)						DESCRIPTION SAMPLE LOCATION (or other field description)	SAS ONLY SPECIFY ADDITIONAL SAS ANALYSES (parameters)
	(A) SOIL OR SEDIMENT	(B) AQUEOUS	(C) EQUIP RINSATE (ORG SOLV)	(D) OTHER (SAS ONLY)	(E) SAMPLE TO SPIKE (check one)	(F) SAMPLE TO DUPLICATE (check one)		
DD011201	X						D01-1	
DD011202	X						D01-2	
DD011203	X						D01-3	
DD011204	X						D02-1	
DD011205	X						D02-1A	
DD011206			X				D02-2	
DD011207	X						D02-3	
DD011208	X				X		D03-1	
DD011209	X						D03-2	
DD011210	X						D03-3	
DD011211	X						D03-4	
DD011212	X					X	D04-1	
DD011213	X						D04-2	
DD011214	X						D05-1	
DD011215	X						D05-2	
DD011216	X						D06-1	
DD011217	X						D06-2	
DD011218	X						D06-3	
DD011219	X						D07-1	
DD011220	X						D07-2	
DD011221	X						D08-1	
DD011222		X			X		DA9-1	
DD011223		X				X	DA9-2	
DD011224		X					DA9-3	

WHITE 540 Copy

YELLOW Client Copy

PINK Lab Copy for Return to 540

GOLD Lab Copy

U.S. ENVIRONMENTAL PROTECTION AGENCY  
 CLP Sample Management Office  
 P.O. Box 818 - Alexandria, Virginia 22313  
 Phone: 703/557-2490 - FTS/557-2490

SAS Number

**SPECIAL ANALYTICAL SERVICE  
 PACKING LIST**

Sampling Office: _____ Sampling Contact: _____ (name) _____ (phone)	Sampling Date(s): _____ Date Shipped: _____ Site Name/Code: _____	Ship To:    Attn:	For Lab Use Only  Date Samples Rec'd: _____ Received By: _____
---	--	-------------------------------	---

Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1. _____	_____	_____
2. _____	_____	_____
3. _____	_____	_____
4. _____	_____	_____
5. _____	_____	_____
6. _____	_____	_____
7. _____	_____	_____
8. _____	_____	_____
9. _____	_____	_____
10. _____	_____	_____
11. _____	_____	_____
12. _____	_____	_____
13. _____	_____	_____
14. _____	_____	_____
15. _____	_____	_____
16. _____	_____	_____
17. _____	_____	_____
18. _____	_____	_____
19. _____	_____	_____
20. _____	_____	_____

For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy

U.S. ENVIRONMENTAL PROTECTION AGENCY  
 CLP Sample Management Office  
 P.O. Box 818 - Alexandria, Virginia 22313  
 Phone: 703/557-2490 - FTS/557-2490

SAS Number  
 1000 - A

SPECIAL ANALYTICAL SERVICE  
 PACKING LIST

Sampling Office: <u>Region I</u>	Sampling Date(s): <u>11/2 - 11/4/88</u>	Ship To: <u>SAS LAB</u>	For Lab Use Only  Date Samples Rec'd:  Received By:
Sampling Contact: <u>Joe Sampler</u> (name)	Date Shipped: <u>11/4/88</u>	<u>100 Main Street</u>	
<u>617/555-1234</u> (phone)	Site Name/Code: <u>#01</u>	<u>Anytown, CO 98765</u>	
		Attn: <u>Jim Smith</u>	

Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1. <u>1000A - 01</u>	<u>LOW CONC. Water - 2,4-D; 2,4,5-TP</u>	
2. <u>1000A - 02</u>	<u>" "</u>	
3. <u>1000A - 03</u>	<u>" "</u>	
4. <u>1000A - 04</u>	<u>" "</u>	
5. <u>1000A - 05</u>	<u>" "</u>	
6. <u>1000A - 06</u>	<u>" "</u>	
7.		
8.		
9.		
10.		
11.		
12.		
13.		
14.		
15.		
16.		
17.		
18.		
19.		
20.		

For Lab Use Only

White - SMO Copy, Yellow - Region Copy, Pink - Lab Copy for return to SMO, Gold - Lab Copy





## CHAIN OF CUSTODY RECORD

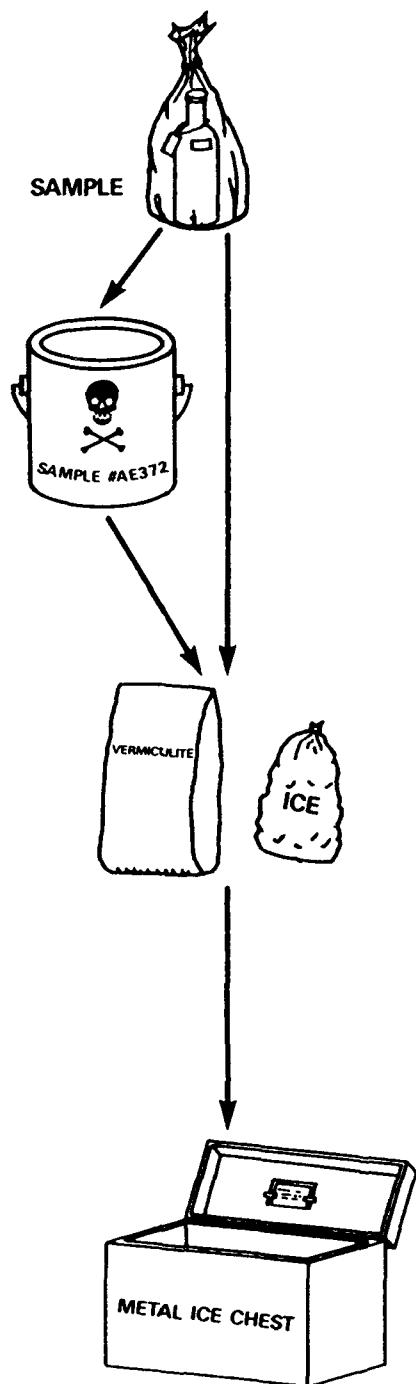
Distribution Original Accompanies Shipment; Copy to Coordinator Field Files

## CHAIN OF CUSTODY RECORD

**Distribution** Original Accompanies Shipment; Copy to Coordinator Field Files

## **SAMPLE PACKAGING AND SHIPMENT**

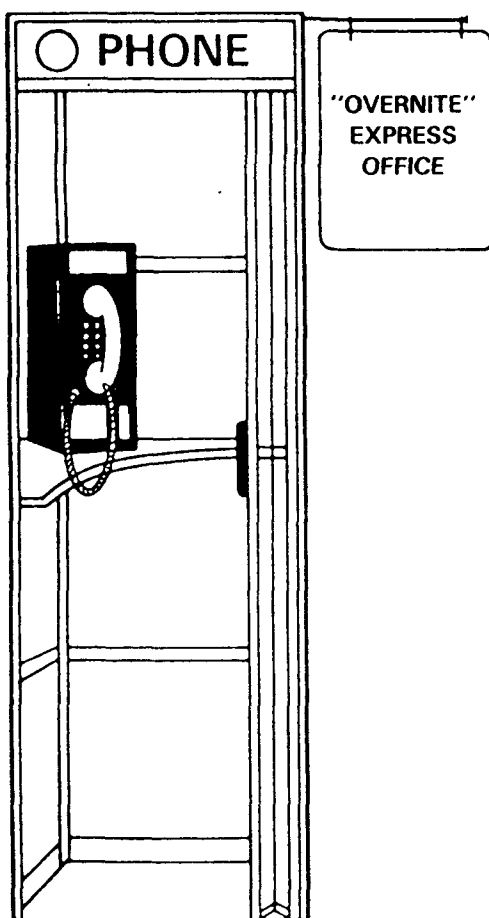
# SAMPLE PACKAGING SUMMARY



- ENCLOSE ALL SAMPLE CONTAINERS IN CLEAR PLASTIC BAGS.
- PACK ALL MEDIUM AND HIGH LEVEL WATER AND SOIL SAMPLES IN METAL PAINT CANS.
- LABEL PAINT CANS WITH SAMPLE NUMBER OF SAMPLE CONTAINED INSIDE.
- SURROUND CONTENTS OF CAN WITH NON-COMBUSTIBLE, ABSORBENT PACKING MATERIAL.
- USING FREEZER PACKAGES OR ICE SEALED IN PLASTIC BAGS, COOL ORGANIC LOW OR MEDIUM SAMPLES AND INORGANIC SAMPLES TO BE ANALYZED FOR CYANIDE TO 4°C.
- ICE IS NOT REQUIRED IN SHIPPING LOW LEVEL SOIL SAMPLES, BUT MAY BE UTILIZED AT THE DISCRETION OF THE SAMPLER.
- DO NOT COOL DIOXIN, INORGANIC LOW LEVEL WATER, INORGANIC MEDIUM/HIGH LEVEL WATER OR SOIL, OR ORGANIC HIGH LEVEL WATER OR SOIL SAMPLES.
- PACK SEALED PAINT CANS OR PLASTIC-ENCLOSED SAMPLE BOTTLES IN SHIPMENT CONTAINER.
- USE A METAL ICE CHEST FOR SHIPMENT (DO NOT USE CARDBOARD OR STYROFOAM CONTAINERS TO SHIP SAMPLES).
- SURROUND CONTENTS WITH NON-COMBUSTIBLE, ABSORBENT PACKING MATERIAL (DO NOT USE EARTH OR ICE PACKING MATERIALS).
- TAPE PAPERWORK IN PLASTIC BAGS UNDER COOLER LID.
- CLOSE COOLER AND SEAL WITH CUSTODY SEALS.

# **SAMPLE SHIPMENT COORDINATION CHECKLIST**

**IMMEDIATELY UPON SHIPMENT OF SAMPLES, SAMPLERS  
CALL SMO AT (703/557-2490), WITH THE FOLLOWING  
INFORMATION:**



- CASE AND/OR SAS NUMBER
- NAME OF LABORATORY
- DATE OF SHIPMENT
- CARRIER, AIRBILL (SHIPMENT) NUMBERS AND TYPE OF SERVICE
- NUMBER AND MATRICES (WATERS, SOILS, ETC.) OF SAMPLES SHIPPED
- INFORMATION ON COMPLETIONS, CHANGES, DELAYS, CONTINUATIONS, ETC., PERTINENT TO THE CASE
- SAMPLER'S NAME, REGION, AND PHONE NUMBER
- SMO MUST BE NOTIFIED BY 3:00 PM ON FRIDAY FOR SAMPLES INTENDED FOR SATURDAY DELIVERY/PICKUP

**POTENTIAL PROBLEMS  
WITH SAMPLE SHIPMENT AND ANALYSIS**

- o **INCORRECT OR INCOMPLETE PAPERWORK**
- o **LABORATORY RECEIPT OF INCORRECT SAMPLES**
- o **INSUFFICIENT VOLUME FOR ANALYSIS REQUESTED**
- o **BROKEN OR LEAKING SAMPLES**
- o **MATRICES OTHER THAN WATER OR SOIL  
(I.E., ROCKS, LEAVES, STICKS, OIL, ETC.)**
- o **NON-HOMOGENEOUS/MULTI-PHASE  
WATER OR SOIL SAMPLES**
- o **ANALYTICAL PROBLEMS WITH SAMPLES**
- o **LABORATORY ACCIDENTS INVOLVING SAMPLES**

**IF ANY OF THESE PROBLEMS ARE ENCOUNTERED,  
CONTACT SMO IMMEDIATELY**

In Reference to Case No(s):

**Contract Laboratory Program  
REGIONAL/LABORATORY COMMUNICATION SYSTEM**

**Telephone Record Log**

Date of Call: \_\_\_\_\_

Laboratory Name: \_\_\_\_\_

Lab Contact: \_\_\_\_\_

Region: \_\_\_\_\_

Regional Contact: \_\_\_\_\_

Call Initiated By: \_\_\_\_ Laboratory \_\_\_\_ Region

In reference to data for the following sample number(s):

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Summary of Questions/Issues Discussed:

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Summary of Resolution:

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

Signature \_\_\_\_\_

Date \_\_\_\_\_

Distribution: (1) Lab Copy, (2) Region Copy, (3) SMO Copy

## **APPENDIX E**

### **AUXILIARY SUPPORT SERVICES DOCUMENTATION**



**PAGE:**

**REGION:**

AS OF

[illegible]

U.S ENVIRONMENTAL PROTECTION AGENCY  
 CONTRACT LABORATORY PROGRAM  
 AS QP

PAGE:

L A B O R A T O R Y   S A M P L E   B A C K L O G   S T A T U S   S U M M A R Y   R E P O R T

LABORATORY:

CONTRACT TYPE:

CONTRACT NO:

CASE NUMBER	SDG NUMBER	SAMPLE NUMBER	SAMPLE SUFFIX	DUE DATE	DATA RECEIPT DATE	DAYS LATE	COMPLETE DATE	STATUS
-----	-----	-----	-----	-----	-----	-----	-----	-----

TOTAL NUMBER OF OUTSTANDING SAMPLES:  
 TOTAL NUMBER OF SAMPLES LATE:  
 TOTAL NUMBER OF SAMPLES INCOMPLETE:

## **CASE FILE PURGE MATERIALS**

**INCLUDE, BUT ARE NOT LIMITED TO:**

**SAMPLE TAGS**

**CHAIN-OF-CUSTODY RECORDS**

**COPIES OF SAMPLE TRACKING RECORDS**

**ANALYSTS' LOGBOOK PAGES**

**INSTRUMENT LOGBOOK PAGES  
(INCLUDING INSTRUMENT CONDITIONS)**

**BENCH SHEETS**

**INSTRUMENT READOUT RECORDS**

**COMPUTER PRINTOUTS**

**CHROMATOGRAPHIC CHARTS**

**RAW DATA SUMMARIES**

**CORRESPONDENCE MEMOS**

**DOCUMENT INVENTORY**

COST RECOVERY DOCUMENTATION CHECKLIST

DATE: OF CHECKLIST: \_\_\_\_\_  
RECEIVED IN HQ \_\_\_\_\_  
COSTS THRU; MONTH \_\_\_\_\_ YEAR \_\_\_\_\_  
REQUIRED DATE FOR REGION \_\_\_\_\_

1. SITE NAME: \_\_\_\_\_ CITY/COUNTY \_\_\_\_\_ STATE \_\_\_\_\_

SITE ID NUMBER: \_\_\_\_\_ NPL \_\_\_\_\_ YES \_\_\_\_\_ NO \_\_\_\_\_

(OTHER NAMES USED FOR THIS SITE): \_\_\_\_\_

2. STATUS: CHECK ONE:

_____ TRIAL DATE (DATE: _____)	_____ IN PREPARATION IN REGION FOR _____
_____ IN DISCOVERY (DEADLINE: _____)	_____ STATUTE OF LIMITATIONS
_____ FILED	_____ PROJECTED/ON GOING NEGOTIATIONS
_____ REFERRED TO DOJ	_____ DEMAND LETTER TO BE SENT
_____ REFERRED TO HEADQUARTERS	_____ ON SCAP
_____ BANKRUPTCY _____	

3. NAME AND TELEPHONE NUMBER OF OSC/REGIONAL CONTACT: \_\_\_\_\_

4. NAME AND TELEPHONE NUMBER OF REGIONAL COUNSEL CONTACT: \_\_\_\_\_

5. WHICH, IF ANY, OF THE FOLLOWING FIT CONTRACTORS WERE USED?

A. E&E (CONTRACT NO. \_\_\_\_\_) \_\_\_\_\_ DATES OF WORK \_\_\_\_\_

B. NUS (CONTRACT NO. \_\_\_\_\_) \_\_\_\_\_ DATES OF WORK \_\_\_\_\_

C. CH<sub>2</sub>M Hill SUBCONTRACTOR E&E, (CONTRACT NO. \_\_\_\_\_) (ZONE II)

DATES OF WORK \_\_\_\_\_

LIST ALL KNOWN TDDs: \_\_\_\_\_

6. WHICH IF ANY OF THE FOLLOWING TAT CONTRACTORS WERE USED?

A. E&E (CONTRACT NO. \_\_\_\_\_) \_\_\_\_\_ DATES OF WORK \_\_\_\_\_

B. ROY F. WESTON (CONTRACT NO. \_\_\_\_\_) \_\_\_\_\_ DATES OF WORK \_\_\_\_\_

LIST ALL KNOWN TDDs: \_\_\_\_\_

7. WAS WORK DONE THROUGH THE CONTRACT LAB PROGRAM (VIAR)? \_\_\_\_ YES \_\_\_\_ NO

A. CONTRACT NO. \_\_\_\_\_ YES \_\_\_\_ NO

B. CONTRACT NO. \_\_\_\_\_ YES \_\_\_\_ NO

C. CONTRACT NO. \_\_\_\_\_ YES \_\_\_\_ NO

IF YES, PLEASE PROVIDE ANY SPECIAL ANALYTICAL SERVICES (SAS) CASE NUMBERS:

\_\_\_\_\_  
\_\_\_\_\_

COST RECOVERY DOCUMENTATION CHECKLIST, PAGE 2

WAS LAB WORK OTHER THAN THROUGH VIAR USED? \_\_\_\_ YES \_\_\_\_ NO

IF YES, PLEASE GIVE LAB NAME AND CONTRACT NUMBER:

\_\_\_\_\_

8. WHICH IF ANY OF THE FOLLOWING REM CONTRACTORS WERE USED?  
(DESCRIBE TASKS WITH THE FOLLOWING: RAMP, IHM, RI/FS, DESIGN  
CONSTRUCTION, COMMUNITY RELATIONS, ENFORCEMENT, OR OTHER)

A. BLACK & VEATCH (CONTRACT NO: \_\_\_\_\_ ) \_\_\_\_\_

DATES OF WORK \_\_\_\_\_ TASK \_\_\_\_\_

B. CAMP DRESSER & MCKEE (CDM) (CONTRACT NO. \_\_\_\_\_ ) \_\_\_\_\_

DATES OF WORK \_\_\_\_\_ TASK \_\_\_\_\_

C. ROY F. WESTON (CONTRACT NO. \_\_\_\_\_ ) \_\_\_\_\_

DATES OF WORK \_\_\_\_\_ TASK \_\_\_\_\_

D. NUS (ZONE I, CONTRACT NO. \_\_\_\_\_ ) \_\_\_\_\_

DATES OF WORK \_\_\_\_\_ TASK \_\_\_\_\_

E. CH<sub>2</sub>M HILL (ZONE II, CONTRACT NO. ( \_\_\_\_\_ ) \_\_\_\_\_

DATES OF WORK \_\_\_\_\_ TASK \_\_\_\_\_

F. CAMP DRESSER MCKEE (REM II CONTRACT NO. \_\_\_\_\_ ) \_\_\_\_\_

DATES OF WORK \_\_\_\_\_ TASK \_\_\_\_\_

G. EBASCO (REM III CONTRACT NO. \_\_\_\_\_ ) \_\_\_\_\_

DATES OF WORK \_\_\_\_\_ TASK \_\_\_\_\_

H. CH<sub>2</sub>M Hill ( REM IV CONTRACT NO. \_\_\_\_\_ ) \_\_\_\_\_

DATES OF WORK \_\_\_\_\_ TASK \_\_\_\_\_

9. PLEASE PROVIDE THE FOLLOWING INFORMATION ABOUT CONTRACTORS  
LET BY AN OSC OR EMERGENCY REMOVAL CLEANUP (ERCS) CONTRACT:

CONTRACTOR: \_\_\_\_\_

CONTRACT NO. \_\_\_\_\_ DELIVERY ORDER No. \_\_\_\_\_

DATES OF WORK: \_\_\_\_\_

10. WAS WORK DONE BY EMERGENCY RESPONSE TEAM (EDISON LAB) \_\_\_\_ YES \_\_\_\_ NO

PAYROLL & TRAVEL COSTS ONLY

DATES OF WORK: \_\_\_\_\_

COST RECOVERY DOCUMENTATION CHECKLIST, PAGE 3

11. WAS WORK DONE THROUGH EERU CONTRACT WITH IT CORP?
- A. Mason & Harger (CONTRACT NO. \_\_\_\_\_) \_\_\_\_ YES \_\_\_\_ NO \_\_\_\_
- B. IT CORP. (CONTRACT NO. \_\_\_\_\_) \_\_\_\_ YES \_\_\_\_ NO \_\_\_\_
- C. (F.W.) Enviresponse Inc.(CONTRACT NO. \_\_\_\_\_) \_\_\_\_ YES \_\_\_\_ NO \_\_\_\_
- DATES OF WORK: \_\_\_\_\_
12. WERE ANY OVERFLIGHTS DONE? \_\_\_\_ YES \_\_\_\_ NO
- DATES OF OVERFLIGHTS: \_\_\_\_\_
13. WAS WORK DONE BY NEIC? \_\_\_\_ YES \_\_\_\_ NO
- DATES OF WORK \_\_\_\_\_ TASK \_\_\_\_\_
14. WAS AN EVIDENCE AUDIT OR OTHER WORK DONE THROUGH NEIC CONTRACT?
- A. With Intera (CONTRACT NO. \_\_\_\_\_) \_\_\_\_ YES \_\_\_\_ NO
- B. WITH TECH LAW (CONTRACT NO. \_\_\_\_\_) \_\_\_\_ YES \_\_\_\_ NO
- C. WITH TECH LAW (CONTRACT NO. \_\_\_\_\_) \_\_\_\_ YES \_\_\_\_ NO
- DATES OF WORK \_\_\_\_\_
- D. FRED C. HART (CONTRACT NO. \_\_\_\_\_) \_\_\_\_\_ or CONTRACT NO. (
15. WAS ANY WORK DONE UNDER THE TES I CONTRACT? \_\_\_\_ YES \_\_\_\_ NO
- A. CONTRACT NO. \_\_\_\_\_ (PRIME CONTRACTOR: GCA)
- DATES OF WORK: \_\_\_\_\_ TASKS PERFORMED: \_\_\_\_\_
- B. WAS ANY WORK DONE UNDER THE TES II CONTRACT? \_\_\_\_ YES \_\_\_\_ NO
- CONTRACT NO. \_\_\_\_\_ (PRIME CONTRACTOR: PRC)
- DATES OF WORK: \_\_\_\_\_ TASKS PERFORMED: \_\_\_\_\_
- C. WAS ANY WORK DONE UNDER TES III CONTRACT? \_\_\_\_\_
- CONTRACT NO. \_\_\_\_\_ (PRIME CONTRACTOR: CDM)
- DATES OF WORK: \_\_\_\_\_ TASKS PERFORMED: \_\_\_\_\_
16. WAS ANY WORK DONE UNDER THE PRE-TES CONTRACT? \_\_\_\_ YES \_\_\_\_ NO
- A. LIFE SYSTEMS (CONTRACT NO. \_\_\_\_\_) \_\_\_\_ YES \_\_\_\_ NO
- B. A.T. KEARNEY (CONTRACT NO. \_\_\_\_\_) \_\_\_\_ YES \_\_\_\_ NO
- DATES OF WORK: \_\_\_\_\_
- NAME ANY OTHER CONTRACTOR USED: \_\_\_\_\_
- CONTRACT NO. \_\_\_\_\_ DATES OF WORK: \_\_\_\_\_

COST RECOVERY DOCUMENTATION CHECKLIST, PAGE 4

17. PLEASE PROVIDE THE FOLLOWING INFORMATION ABOUT OTHER FEDERAL AGENCIES THAT WORKED ON THE SITE:

AGENCY	IAG #	DATES OF WORK	CONTACT PERSON/TELEPHONE
HHS			
COE			
USCG			
FEMA			
DOJ			
DOI			
NOAA			
USGS			

BRIEF DESCRIPTION OF WORK:

\_\_\_\_\_

\_\_\_\_\_

18. WAS THERE A STATE COOPERATIVE AGREEMENT OR CONTRACT? ☐ YES ☐ NO

STATE: \_\_\_\_\_ COOPERATIVE AGREEMENT # \_\_\_\_\_

CONTRACT No. \_\_\_\_\_

19. WERE ANY OTHER CONTRACTORS (e.g., R&D CONTRACTS) USED? IF SO, PLEASE PROVIDE THE FOLLOWING:

CONTRACTOR: \_\_\_\_\_

CONTRACT No: \_\_\_\_\_

DATES OF WORK: \_\_\_\_\_

BRIEF DESCRIPTION OF WORK: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

COST RECOVERY DOCUMENTATION CHECKLIST, PAGE 5

20. WERE ANY REGIONAL COUNSEL APPROPRIATIONS FOR LEGAL EXPENSES  
USED?    ☐ YES    ☐ NO

21. PLEASE LIST THE REGIONAL OFFICES WHICH HAVE BEEN INVOLVED IN THE CASE:

\_\_\_\_\_

22. ANY OTHER PERTINENT INFORMATION NOT PROVIDED ABOVE:

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_



**MEMORANDUM**

**DATE:** \_\_\_\_\_  
**TO:** Data Review Team  
Sample Management Office  
**FROM:** \_\_\_\_\_  
USEPA Region \_\_\_\_\_  
**SUBJECT:** Data Review Request  
**COPIES:**

Please review the data from the following SMO Case:

SMO Case No.: \_\_\_\_\_  
Site Name: \_\_\_\_\_  
Lab Name(s): \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**I. Sample Information:**

- A. Number of Samples in Case: \_\_\_\_\_  
B. Number of Samples to be Reviewed: \_\_\_\_\_

(List Numbers if Not All)

_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____
_____	_____	_____	_____

- C. Organics to be Reviewed? Yes\_\_\_ No\_\_\_  
D. Inorganics to be Reviewed? Yes\_\_\_ No\_\_\_

**II. User Information:**

A. User Organization: \_\_\_\_\_

B. Contact for Questions:

Name: \_\_\_\_\_ Telephone: \_\_\_\_\_

C. Type(s) of Review Requested:

	<u>Check All That Apply</u>	<u>Date Needed</u>
QA/QC Compliance	<input type="checkbox"/>	_____
Problem Case	<input type="checkbox"/>	_____
Applications	<input type="checkbox"/>	_____
Consulting	<input type="checkbox"/>	_____
Other	<input type="checkbox"/>	_____

Specify: \_\_\_\_\_

D. Additional Issues to Address in Review: \_\_\_\_\_

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

E. Intended Use of Data:

	<u>Check All That Apply</u>
Enforcement	<input type="checkbox"/>
Preliminary Assessment	<input type="checkbox"/>
Site Investigation	<input type="checkbox"/>
Remedial Action	<input type="checkbox"/>
Site Monitoring	<input type="checkbox"/>
Undetermined	<input type="checkbox"/>
Other	<input type="checkbox"/>

Specify: \_\_\_\_\_

\_\_\_\_\_

F. Comments: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

## **CONTRACT COMPLIANCE SCREENING**





**CONTRA**

**CASE:**

LAB NAME:

[illegible]







312

55-6734-10

[illegible]



**CONTRA**

SE NUMBER :

**B NAME :**

CONTRACT NO.:

[illegible]



## **APPENDIX F**

### **REFERENCES**

**NOTE:** The references in this appendix are supplied for general information purposes and do not necessarily represent methods or procedures utilized in the CLP.

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